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DOCUMENT-IDENTIFIER: US 6190560 B1

TITLE: Slurry fill method and valve

DATE-ISSUED: February 20, 2001

US-CL-CURRENT: 210/656; 210/198.2

APPL-NO: 09/ 410156

DATE FILED: September 30, 1999

---------- KWIC ---------

Abstract Text - ABTX:

A chromatography column has valve and method for filling it with a slurry of media, the valve utilizing a **piston** to compact the slurry in the column. The **piston** moves through a cavity in communication with both a slurry inlet and slurry ports which are in communication with the interior of the chromatography column. The **piston** assists in compacting the slurry within the column. The method of filling the column does not require disassembly of the column. The **piston** may also be utilized to change the valve position from a slurry fill position to a normal operation position or other position.

Brief Summary Text - BSTX:

It is yet another object of the present invention to provide a **piston** to alter the operational status of a valve assembly.

Brief Summary Text - BSTX:

Accordingly, the present invention provides a chromatography column having a media and a product inlet assembly positioned at the inlet of a nozzle-type assembly at the top of the column, the media and product inlet assembly preferably including a housing having a product/buffer inlet passage and a media inlet passage extending through the housing. A **piston** having a pressurized fluid source is preferably connected to the housing to assist in the filling, distributing, and/or packing of the media bed within the column. Additionally, the **piston** may control the operational status of the slurry inlet assembly. A similar, or the same, valve may be utilized to remove slurry from a chromatography column as well.

Detailed Description Text - DETX:

FIG. 3 depicts the media filling valve assembly 100 in a first position. This position may be referred to as the pack position or the slurry into column position. The valve assembly 100 is illustrated having a fluid supply, shown as a hydraulic or air cylinder 102, connected to a **piston** 104 by a connecting rod 106. The air or hydraulic cylinder 102 **drives** the connecting rod 106 to move the **piston** 104.

Detailed Description Text - DETX:

The first position has the **piston** 104 obscuring flow from the slurry inlet 108 to a clean in place port 110. This first position may be referred to as the packing position. The **piston** 104 is preferably located just above the slurry inlet 108 when the valve assembly 100 is in the first position. Slurry and/or media may be pumped into the column 10 through at least one, and preferably a

plurality, slurry spray ports 112 in an assembly housing 116. More specifically, slurry enters the valve assembly 100 from a supply of slurry at the slurry passage, or slurry inlet 108. From the slurry inlet 108, slurry may proceed into a cavity 114 of the valve assembly 100. Once in the cavity 114, the **piston** 104 obscures flow to the clean in place port 110. Accordingly, slurry will flow through the cavity 114 to the slurry port or ports 112. The slurry ports 112 are preferably multi-port spray holes, but depending on the particular application, the slurry ports 112 may transfer slurry to one or more other distribution devices.

Detailed Description Text - DETX:

When the column 10 is full of slurry during a filling operation, the operator will be alerted since the pump pumping the slurry in will stall. At this point, the operator may stop the source of slurry into the slurry inlet 108. Looking to FIG. 4, the valve assembly 100 may then be operated such that the **piston** 104 is moved to the second position. The second position may also be referred to as the run position.

Detailed Description Text - DETX:

The **piston** 104 is illustrated as being at or near the distribution cap 126, however this need not necessarily be the case. The fluid source is utilized to pack the slurry in the column 10 when the valve assembly 100 is in the second position by pushing slurry remaining in the cavity 114 into the column through the slurry port or ports 112. Preferably, the **piston** 104 is stroked from its location above the slurry inlet 108 to a position below the slurry inlet 108.

Most preferably, the **piston** 104 is stroked to at or near the distribution cap 116. As the **piston** 104 is driven by the cylinder 102 towards the second position, the slurry is compacted in the column 10. As the **piston** 104 moves through the cavity 114, the slurry will be pushed out the slurry port, or ports 112. This will assist in compacting the slurry in the column 10.

Detailed Description Text - DETX:

The **piston** 104 is shown with **piston** rings 132 which may assist in minimizing, and preferably preventing any fluid flow from the column 10 past the **piston** 114. For some applications, the valve assembly may be stroked to the second position and back to the first position a number of times in order to force more slurry into the column 10. This process is believed to provide a more compressed bed of resin in the column 10.

Detailed Description Text - DETX:

While the valve assembly 100 is in the second position, the clean in place port 110 may be open. This will allow the valve a portion of the cavity 114 of the valve assembly 100 to be washed clean. Additionally an open clean in place port may prepare the valve assembly 100 for retracting the **piston** 104 when necessary. The clean in place port 110 may be open to assist in the movement of the **piston** 104 from the second to the first or third position. At least in the movement from the second to the first position, an open clean in place port 110 will provide an outlet for any slurry or other material in the cavity 114 or elsewhere in the valve assembly 100. It is also possible that the slurry inlet 108 may be left open to assist in the stroking of the

valve assembly from
the second to the third or first positions.

Detailed Description Text - DETX:

FIG. 5 shows the valve assembly 100 in the third position. Some applications
may find this placement of the **piston** 104 useful to assist in cleaning the
interior or cavity 114 of the valve assembly 100. The clean in place port 110
is in fluid communication with the slurry inlet 108, and at least a portion of
the cavity 114 may be cleaned with the valve assembly 100 in this position.

Detailed Description Text - DETX:

FIG. 6, which is very similar to FIG. 3., depicts the valve assembly utilized
as a slurry outlet valve assembly 200. The valve assembly 200 is illustrated
having a fluid supply, shown as hydraulic or air cylinder 202, connected to a
piston 204 by a connecting rod 206. The air or hydraulic cylinder 202 **drives**
the connecting rod 206 to move the **piston** 204.

Detailed Description Text - DETX:

The operation of the slurry outlet valve 200 again is very similar to that of
the media filling valve assembly 100. The position shown in FIG. 6 is the
slurry out position. Slurry may be removed from a chromatography column 10
utilizing slurry outlet valve 200. Inlet port 212 provides access to the
slurry passage, or slurry outlet 208, for slurry to be removed from the
chromatography column 10 when the **piston** 204 is in the position shown. If the
piston 204 is moved closer to the inlet port 212 such that the slurry outlet
208 is no longer in communication with the inlet port 212,

the slurry outlet valve 200 will be in a normal operating position. If the **piston** 204 is positioned closer to the cylinder 202 such that a clean in place port 210 is in communication with the slurry outlet 208, the slurry outlet valve 200 will be in a clean in place position. The operation of the slurry outlet valve in the preferred embodiment may be substantially similar to that of the slurry inlet valve 100 as aforesaid or may be utilized in other ways which would be obvious to one skilled in the art.

Claims Text - CLTX:

a) providing a slurry inlet valve having a cavity and a **piston** at least partially within the cavity, said valve communicating through said cavity with an interior volume of the column;

Claims Text - CLTX:

d) operating said **piston** from a first position to a second position within at least a portion of the cavity to force at least some of the slurry from the cavity into the interior volume of the column;

Claims Text - CLTX:

e) returning the **piston** from the second position towards the first position;

Claims Text - CLTX:

h) operating said **piston** from about the first position towards the second position to force at least some of the slurry from the cavity into the interior volume of the column.

Current US Original Classification - CCOR:

210/656

Current US Cross Reference Classification - CCXR:

210/198.2

US-PAT-NO: 6001260
DOCUMENT-IDENTIFIER: US 6001260 A

TITLE: Collapsible apparatus for compressing packing material in liquid chromatography columns and methods of use

DATE-ISSUED: December 14, 1999

US-CL-CURRENT: 210/656; 210/198.2

APPL-NO: 08/ 888252

DATE FILED: June 3, 1997

PARENT-CASE:

RELATED APPLICATION This Application is a division of U.S. patent application Ser. No. 08/609,976 filed Feb. 29,1996, now U.S. Pat. No. 5,667,675.

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Abstract Text - ABTX:

Longitudinally collapsible apparatus for compacting packing material within a tubular liquid chromatography column. The apparatus includes a first support member, a second support member and a movable support member. The movable support member is releasably connectable to the second support member for support thereby. When released from the second support member, the movable member is adapted to ride on guide members extending between the first and second support members. A compression mechanism is supported on the movable support member to move therewith to a longitudinally collapsed position for the apparatus. The compression mechanism comprises a

reversible **drive** for an
axially movable rod carrying a movable **piston**. With the movable support member
secured to the second support member, the **piston** of the compression mechanism
is movable in an end of the tubular column when the column is supported on the
first support member. Movement of the movable **piston** within the column axially
compresses a column packing material as the **piston** is driven towards an
opposite end of the column. The **piston** also is releasably connectable to the
tubular column to raise and lower the column when the apparatus extends in a
vertical direction and is it is desired to insert an end **piston** into or remove
the end **piston** from the opposite end of the column or when it is desired to
change an adapter ring on the first support member to receive a column of a
different diameter for packing by the apparatus.

Brief Summary Text - BSTX:

Most preparative chromatography is performed in tubular metal columns of 2, 4
or 6 inch or larger diameter. Preparation of such large diameter columns is
difficult using conventional slurry packing wherein a suspension of stationary
phase is forced or "packed" into a chromatographic column using a high pressure
stream of liquid to produce a homogeneous and stable column structure. Rather,
today, the preferred method of preparing large diameter columns for use in
preparative chromatography is the method commonly referred to as "axial
compression". Axial compression is described in U.S. Pat. Nos. 3,466,809
and 5,169,522. In the '809 patent a **piston** containing a filter matrix and
fluid sealing mechanism is used to compact a bed of stationary phase and
maintain adequate compression to correct column

instability. In the '522patent a compression ram is used to consolidate the stationary phase during the packing operation.

Brief Summary Text - BSTX:

The present invention provides apparatus for producing and operating highly efficient preparative columns while avoiding the disadvantages of the prior art. In the present invention, a tubular column or column tube is provided with end closures containing filters capable of retaining the stationary phase and provisions for the introduction and removal of liquid under pressure. The end closures, generally known as bed supports, are supported within opposite ends of the column tube against outward axial motion by **piston** elements which provide both fluid connections for the passage of mobile phase and high pressure sealing elements to prevent leakage of the mobile phase from the column. At least one of the bed supports and its supporting **piston** are movable within the column tube and are subjected by means of a compression mechanism to a compressive force larger than that generated by the pressure drop occurring from the passage of liquid through the stationary phase. The compressive force permits the preparation and continued operation of high performance liquid chromatographic columns.

Brief Summary Text - BSTX:

In a typical embodiment of the invention, the compressive mechanism comprises a hydraulic press including a vertically extending hydraulic cylinder containing a compression rod supporting a compression **piston** at its lower end. The compression **piston** functions as the upper support **piston**

for the column tube containing packing material to be compressed by the apparatus of the present invention. The cylinder is mounted on a movable support member which is mounted for vertical movement on vertical guide members extending between upper and lower support members. The lower support member is designed to support the column tube containing packing material which is to be compressed. The movable support member is designed for releasable connection to the upper support member. When the movable member is released from the upper support member it is movable downward to vertically collapse the apparatus of the present invention. When the movable support member is connected to the upper support member, the compression **piston** is movable axially into an upper open end of the column tube which is vertically mounted on the lower support member. Upon actuation of the compression mechanism, the compression rod is driven downward to **drive** the compression **piston** into the column tube to engage an upper bed support and to compact the packing material within the column tube against a lower bed support and a stationary lower **piston** within a lower end of the column tube. The compression **piston** and the lower stationary **piston** include annular fluid seals for sealing with an inner surface of the column tube and contain fluid passages for the introduction and removal of mobile phase. The lower support member is provided with an opening through which spent packing may be extruded. This opening is reduced in diameter for packing and operation by the mounting of an adapter plate or ring. The adapter ring may be selected from a variety of diameters to permit the operation of columns of different diameters using the same compression mechanism.

Brief Summary Text - BSTX:

To prepare a liquid chromatography column using the apparatus of the present invention, the correct size adapter ring is mounted on the lower support member. The lower **piston** and bed support are then mounted on the lower support member via the adapter ring, and the column tube is pressed downward onto the lower bed support and lower **piston** to close the lower end of the column tube. A suspension of stationary phase is introduced into the upper end column tube, which is then closed by the upper bed support. At least one of the bed support-**piston** sets is provided with a connection for liquid to exit the apparatus during processing. The upper movable bed support is then subjected to a compressive force by the compression **piston,** which causes the movable bed support to travel downward within the column tube in an axial direction until excess slurry solvent is expelled and the stationary phase is compacted. The column is then ready for operation and may be connected to a liquid chromatographic system for use. There is no need for additional processing operations and the column is suitable for additional compression, unlike those prepared in accordance with U.S. Pat. No. 4,549,584.

Brief Summary Text - BSTX:

In the present invention, the stationary phase in the column is operated under compression by the movable upper **piston**. However, both the upper and lower **pistons** can be removed from the column tube without removing the bed supports. Since there is no need to remove the movable bed supports from the column tube, there is reduced danger of damage to the stationary phase structure from removal of the **pistons**. The compression mechanism and

pistons can then be used to prepare additional columns without destroying the columns previously prepared using the compression mechanism. Also, columns previously prepared using the compression mechanism may be recompressed and again operated under compression to maintain column performance.

Brief Summary Text - BSTX:

It is a further feature of the present invention that the use of interchangeable adapter plates or rings and **piston** sets permits the use of the compression mechanism with columns of differing diameters without encumbering each column with complex and costly flanges or other devices to connect to fluid flow passages. This allows column tubes according to the present invention to be of simple design and constitutes an advantage over previous designs.

Brief Summary Text - BSTX:

After the use of chromatographic column of the present invention is complete, the spent stationary phase is easily removed from the column by raising the column tube, removing the lower **piston,** lowering the column tube and using the compression mechanism to extrude the stationary phase through the opening in the lower support member. The bed supports are easily removed by the same extrusion. The use of the adapter plates and lower **piston** provides for this simple extrusion process, which is not present in the Merck system.

Brief Summary Text - BSTX:

In the present invention, the hydraulic press is adapted to

two further uses
beyond the compression of the stationary phase for
operation or extrusion.
First, the hydraulic press is used to raise and lower the
column tube during
the operations of inserting the lower bed support and
piston, and during the
removal of the **piston** before stationary phase extrusion.
Since large diameter
column tubes can weigh more than 50 pounds even when empty,
such use of the
hydraulic press provides a significant advantage to the
operator. The operator
need only mount the column tube on the lower support of the
apparatus. All
subsequent lifting and lowering is performed by the
apparatus itself. In
particular, such column raising and lowering is
accomplished by coupling the
upper end of the column tube so that the tube follows the
motion of the
compression rod. The coupling can be performed using a
number of methods. The
preferred method is by using a removable pin which passes
through a hole
through the column tube and the **piston** at a right angle to
the tube axis.

Drawing Description Text - DRTX:

FIG. 1a is a an exploded view of a column packing
compression mechanism shown
in FIG. 1 illustrating the component part comprising a
vertically extending
compression rod, a **piston** at a lower end of the compression
rod and a **drive** for
vertically moving the compression rod comprising a
hydraulic cylinder.

Drawing Description Text - DRTX:

FIG. 1b is a side view showing the components in FIG. 1a
assembled with the
piston secured to the compression rod by a universal
coupler and a laterally
extending pin, the **piston** shown in FIGS. 1, 1a and 1b being

a "dummy" **piston**
for use when the apparatus of the present invention is to be vertically
collapsed.

Drawing Description Text - DRTX:

FIG. 2 is a side view partially in section of the apparatus of FIG. 1 with the
moveable support plate secured to an upper support member and with the moveable
support plate supporting the hydraulic cylinder in an upward extended position
with a compression rod of the column packing compression mechanism and "dummy"
piston bearing on the lower support member.

Drawing Description Text - DRTX:

FIG. 3 is a side view partially in section showing an adaptor ring mounted on a
lower support member of the apparatus and vertically supporting a column tube
between the lower support member and the moveable and upper support members.
The compression rod of the column packing compression mechanism is shown
supporting a compression **piston** for fitting into an upper open end of the
column tube. FIG. 3 also depicts a front face of the control panel for
controlling operation of the apparatus.

Drawing Description Text - DRTX:

FIG. 4 is a side view partially in section similar to FIG. 3 and illustrating
the compression rod and compression **piston** releaseably secured to the column
tube by a pin extending through aligned openings in the column tube and
compression rod above the compression **piston**. FIG. 4 shows the compression rod
in a raised position to lift the column tube from the adaptor ring and lower

support member, the adaptor ring shown supporting a lower **piston** and bed
support for closing the lower open end of the column tube. The lower **piston**
includes openings for passing liquid from the column tube during operation of
the column.

Drawing Description Text - DRTX:

FIG. 5 is a side view partially in section of the apparatus of FIG. 4 showing
the column tube after it has been driven downward onto the adaptor ring to
receive the lower **piston** and lower end closure.

Drawing Description Text - DRTX:

FIG. 6 is a side view partially in section of the apparatus of FIG. 5 after the
connecting pin has been removed to release the compression **piston** and
compression rod from the column tube, the compression rod and compression
piston being depicted in a raised position with the upper end of the column
tube open to receive a slurry of the packing material for packing upon a
downward movement of the compression **piston** into the column tube.

Detailed Description Text - DETX:

The apparatus 10 further comprises a column packing compression mechanism 22
comprising a compression rod 24, a compression **piston** 26 and a **drive** 28
preferably including a hydraulic cylinder 30 having lower and upper flanges 32
and 34 respectively secured by tie rods 36. The column packing compression
mechanism 22 is secured to movable support member 18 for vertical movement
therewith to vertically collapse 10 when the moveable support member 18 is

released from the upper support member 12 as depicted in FIG. 1.

Detailed Description Text - DETX:

When the moveable support member 18 is secured to the upper support member 12, operation of the **drive** 28 by actuation of knobs on a control panel 37 cause the compression rod 24 to move vertically to raise and lower the compression **piston** 26 which is sized to fit snugly into an open upper end of a column tube 10 as shown in FIGS. 3, 4, 5 and 6. The column tube is supported on the lower support member 14 over the central hole 15 and downward movement of the compression **piston** 26 within the column tube compresses a packing material within a lower end of the column tube.

Detailed Description Text - DETX:

As previously indicated, the upper support member 12 is adapted to secure the moveable support member 18 thereto when it is desired to operate the apparatus 1 in compressing a packing material within the column tube 10. To accommodate such connection, the support blocks 64 and 66 include through holes 64a and 66a respectively. Such holes are designed to receive vertically extending bolts 64b and 66b passing through the support blocks 64 and 66 into threaded holes 65 moveable support member 18 (see FIG. 1c) to releaseably secure the moveable support member to the upper support member 12 as shown in FIG. 2a. Thus secured to the upper support member 12, the moveable support member 18 provides vertical support for the hydraulic cylinder 30 of the column packing compression mechanism 22 above the upper support member 12 as shown in FIG. 3. In this regard, and as most clearly shown in FIGS. 1c and

1d, the moveable support member 18 comprises a flat plate including a central stepped opening 71 defining an annular shoulder 72 for receiving a lower face of a cylinder gland 73 of the hydraulic cylinder 30. Thus positioned, the lower flange 32 of the cylinder rests on the top face of the moveable support plate 18 with bolts extending through mating threaded holes 74 in the moveable support plate and lower flange 32. The threaded holes 74 receive bolts 75 to releaseably secure the flange to the moveable support plate as shown most clearly in FIG. 1. With the column packing compression mechanism 22 thus supported on the moveable support member 18 as shown in FIG. 1, the compression rod 24 extends axially from the cylinder 30 through the opening 71 in the moveable support plate 18 to releaseably secure the compression **piston** 26 (FIG. 3) in line with an open upper end of the column tube 35 mounted on the lower support member 14 as shown in FIG. 3. Such connection of the compression rod 24 to the compression **piston** 26 is most clearly depicted in FIG. 3 as well as in FIGS. 1a and 1b for a "dummy" **piston** connected to the compression rod 24. As there illustrated, the lower end of the compression rod 24 includes a threaded socket 77 for receiving an externally threaded upper end of a cylindrical coupler 78 to secure the coupler to the compression rod 24. The coupler 78 also include a lower socket 80 having laterally extending side holes there through. The socket 80 is dimensioned to receive the upper end of the compression **piston** 26 in FIG. 3 or a "dummy" **piston** 26a as depicted in FIGS. 1a and 1b. In both instances, the **piston** includes a laterally extending through hole for receiving a connecting pin 82 after the **piston** has been inserted upwardly into the lower socket 80 as most clearly depicted in FIGS. 1b and 3.

Detailed Description Text - DETX:

In the vertically collapsed position for the apparatus 10, the "dummy" **piston** 26a bears adaptor ring 46 mounted on the lower support member 14. In the raised position for the moveable support member 16 as depicted in FIGS. 3-6, the compression system 22 is raised from the lower support member 14 which provides vertical support for the column tube 35 containing packing material which is to be compressed upon downward movement of the compression **piston** 26 into the column tube. To provide such support for the "dummy" **piston** 26a as shown FIGS. 1 and 2 and for the column tube 35 as shown in FIGS. 3-6, the lower support plate 14 includes the previously described adaptor ring 46. As shown most clearly in FIG. 1f, the adaptor ring includes an annular recess 84 forming an annular shoulder 85 for receiving a bottom face of the "dummy" **piston** 26a as depicted in FIGS. 1 and 2 or the lower end of the column tube 35 as depicted in FIGS. 3-6.

Detailed Description Text - DETX:

As previously indicated, user control of the controls on the control panel 37 determines the operation of the apparatus 10 in moving to and from the vertically collapsed position shown in FIG. 1 and in the placement of the column tube 35 and its end closures and **pistons** in the ends of the column tube and the subsequent compression of a packing material within the column. To accomplish this, the column packing compression mechanism 22 as previously described preferably is a model 2H hydraulic ram manufactured by Parker-Hannifin Corp. of Des Plaines, Ill. The hydraulic

ram is connected in a conventional manner and controlled by the fluid circuit depicted in FIG. 7 under control of the controls indicated on the control panel 37. In particular, the ram comprises the hydraulic cylinder 30 with the compression rod 24 extending downwardly therefrom. Fluid to control the raising and lowering of the compression rod within the cylinder is controlled by operation of a directional control valve 90 shown in FIG. 7 and on the control panel 37 in FIGS. 1, 2 and 3 through 6.

Detailed Description Text - DETX:

The directional valve 90 is a conventional three position valve for regulating the direction of fluid flow to and from the hydraulic cylinder 30. The hydraulic cylinder includes an internal **piston** for driving the compression rod 24 in and out of the hydraulic cylinder in a conventional manner. For example, with the directional control valve 90 in the position indicated, fluid flows into an upper end of the hydraulic cylinder 30 and returns from a lower end of the cylinder to **drive** the compression rod 24 downwardly and out of the hydraulic cylinder. When the directional control valve 90 is in its second or intermediate position, all flow of fluid to the hydraulic cylinder 30 is blocked. When the control valve is in its lower or third position, the direction of flow is reversed such that fluid flows into the lower end of the hydraulic cylinder 30 below its internal **piston** and exits the hydraulic cylinder at the upper end for return to the reservoir 92.

Detailed Description Text - DETX:

In this regard, and as previously indicated, the apparatus

10 is susceptible as
several different operations. First, as depicted in FIGS.
1 and 2, the
apparatus may be vertically collapsed to reduce the
vertical dimension of the
apparatus to a minimum allowing for compact packaging and
shipping of the
apparatus as well as for movement of the apparatus from one
location to another
in a laboratory through doorways and the like without
requiring a tilting or
rotation of components of the apparatus. Such collapsing
of the apparatus 10
is accomplished by removing the column tube 35 from the
apparatus and with the
"dummy" **piston** 26a attached to the lower end of the
compression rod 24, setting
the control knob "RAM" to the "lower" position. This
results in the
compression rod being driven downwardly to the position
indicated in FIG. 2
with the "dummy" **piston** bearing on the top of the adaptor
ring 46 secured to
the lower support member 14. Then, upon a disconnection of
the connector 20
securing the moveable support member 18 to the upper
support member 12,
continued operation of the hydraulic system with the RAM
control in the "raise"
position lowers the hydraulic cylinder 30 with the moveable
support member 18
downwardly on the guide rods 16 to the lower or collapsed
position shown in
FIG. 1.

Detailed Description Text - DETX:

When it is desired to again extend the apparatus 10, the
process is simply
reversed. With the hydraulic cylinder 30 in the lower
position shown in FIG.
1, the control knob "RAM" is switched to the "lower"
position. This **drives** the
hydraulic cylinder 30 and moveable support member 18
upwardly to the position
shown in FIG. 2 where the connector 20 again releaseably
locks the moveable

support member to the upper support member. Continued operation of the hydraulic system then with the control knob "RAM" in the "raise" position withdraws the compression rod 24 upwardly within the hydraulic cylinder 30 to the position shown in FIG. 3 where the "dummy" **piston** 26a may be replaced by the compression **piston** 26 and the apparatus 10 readied to receive a column tube 35 as shown in FIG. 3.

Detailed Description Text - DETX:

As shown in FIG. 3, the adaptor ring 46 of proper internal diameter is first secured to the top of the lower support plate 14 and an empty column tube 35 mounted thereon as shown. As depicted in FIG. 4, the compression rod and **piston** are then driven downwardly into an upper end of the empty column tube. Next, the compression rod and **piston** are releaseably secured to the column tube by a connector pin 96 passing through aligned holes 99 in the column tube and the **piston** coupler 98 as shown in FIG. 4. Upward movement of the compression rod and **piston** then lifts the column tube 35 from the adaptor 46 allowing a lower **piston** and bed support 100 including filters and fluid ports to be located on the adaptor ring as illustrated. Next, the compression rod and **piston** are lowered to lower the column tube 35 onto the bed support and lower **piston** 102 as depicted in FIG. 5. This is an important feature of the apparatus 10 of the present invention. Since the large diameter column tubes processed by the apparatus of the present invention are heavy even when empty and difficult to maneuver and position over lower **pistons** and bed supports. Furthermore, insertion of tightly fitting bed supports requires significant

force. With the apparatus of the present invention however, all such lifting and positioning is accomplished by the hydraulic system of the compression mechanism 22.

Detailed Description Text - DETX:

Once the column tube 35 is seated on the lower **piston** and bed support 100 as depicted in FIG. 5, the connecting pin 98 is removed from the column tube 35 and coupler 78 and the compression rod and **piston** are allowed to move upwardly out of the upward end of the column tube to the position shown in FIG. 6. The upper open end of the column tube 35 is then clear to allow a pouring of a slurry of packing material into the column tube for compression by the compression **piston** 26 after an upper bed support is inserted into the column tube.

Detailed Description Text - DETX:

Thus, as described above the present invention overcomes the short comings of prior column packing apparatus by providing an apparatus which is vertically collapsible for packaging, shipment and relocation from one location to another in a laboratory. Also, the apparatus of the present invention provides for the lifting and positioning of empty column tubes and the positioning of lower **pistons** and bed supports therein without requiring operators to manually lift, locate and lower the column tubes. Further, the apparatus of the present invention is adapted to compact columns of different diameter by use of adaptor rings and compression **pistons** of different size which are readily mounted and removed from the apparatus which in packing operation repeatedly uses the same

compression mechanism in the packing of such columns.

Detailed Description Text - DETX:

While a particular embodiment of the apparatus of the present has been described herein, changes and modifications in the illustrated embodiment may be made without departing from the spirit or scope of the present invention. For example, the apparatus may be inverted such that the compression mechanism is mounted below the column tube with the compression **piston** entering a lower open end of the tube to compress packing material as it is driven upward into the tube. Alternatively, the apparatus may be positioned horizontally rather than vertically. Accordingly, it is intended that the scope of the present invention be limited only by the terms of the following claims.

Claims Text - CLTX:

inserting a compression **piston** of a column packing compression mechanism vertically into an upper open end of the first column tube;

Claims Text - CLTX:

releaseably connecting the compression **piston** to the first column tube;

Claims Text - CLTX:

actuating the compression mechanism to raise the compression **piston** and lift the first column tube from the lower support member;

Claims Text - CLTX:

positioning a lower **piston** and column tube end closure on

the adaptor ring;

Claims Text - CLTX:

actuating the compression mechanism to lower the compression **piston** and first column tube onto the end closure, lower **piston** and adaptor ring;

Claims Text - CLTX:

disconnecting the compression **piston** from the first column tube;

Claims Text - CLTX:

raising the compression **piston** from the upper end of the column tube whereby the first column tube is open to receive a slurry of a packing material;

Claims Text - CLTX:

positioning the compression **piston** in the upper end of the first column tube; and

Claims Text - CLTX:

driving the compression **piston** and upper end closure downward to compress the packing material within the first column tube.

Claims Text - CLTX:

releaseably connecting the compression **piston** to an upper end of the first column tube to lift the first column tube;

Claims Text - CLTX:

driving the compression **piston** upward to raise the first column tube from the lower support member;

Claims Text - CLTX:

releasing the compression **piston** from the first column tube to allow the first column tube to be removed from the apparatus;

Claims Text - CLTX:

replacing the compression **piston** with a new compression **piston** having a diameter matching the inner diameter of a second column tube to be packed by the apparatus;

Claims Text - CLTX:

lowering the new compression **piston** into an upper end of a second column tube;

Claims Text - CLTX:

releaseably connecting the new compression **piston** to the second column tube;

Claims Text - CLTX:

raising the new compression **piston** to lift a second column tube from the lower support member;

Claims Text - CLTX:

mounting a lower **piston** and lower end closure having outer matching the second column tube on the new adaptor ring;

Claims Text - CLTX:

lowering the new compression **piston** and second column tube onto the second end
closure and second end **piston**; and

Claims Text - CLTX:

releasing the new compression **piston** from the second column tube to allow a
removal of the new compression **piston** therefrom and entry of a slurry of
packing material into the second column tube for a compression by downward
movement of the new compression **piston** within the second column tube.

Current US Original Classification - CCOR:

210/656

Current US Cross Reference Classification - CCXR:

210/198.2

US-PAT-NO: 5951873
DOCUMENT-IDENTIFIER: US 5951873 A

TITLE: Chromatographic device with **piston** locking mechanism and method of packing same

DATE-ISSUED: September 14, 1999

US-CL-CURRENT: 210/656; 210/198.2

APPL-NO: 08/ 996973

DATE FILED: December 23, 1997

---------- KWIC ---------

TITLE - TI:

Chromatographic device with **piston** locking mechanism and method of packing same

Abstract Text - ABTX:

A chromatographic device comprising a column body having a first end and a closed second end. The device further includes a **piston** inside the column body, a chromatographic medium, a rod, and a locking mechanism. The chromatographic medium is within the column body and between the **piston** and the second end. The rod has a longitudinal rod axis and is operatively engageable with the **piston** for pressing the **piston** in the column body. The locking mechanism is adapted for locking the rod relative to the column body in a manner to prevent movement of the rod relative to the column body in an outward longitudinal direction away from the **piston** without preventing movement of the

rod relative to the column body in an inward longitudinal direction toward the **piston**. Preferably, the chromatographic device includes a spring for exerting a constant axial compression against the **piston** to prevent the formation of voids in the packing medium.

Brief Summary Text - BSTX:

This invention relates to the liquid chromatography, and more particularly to chromatographic devices having **pistons** for compressing a chromatographic medium.

Brief Summary Text - BSTX:

A chromatographic device includes a chromatographic column (having a cylindric column body and a fixed end plate covering one end of the column body), a **piston** slidable within the column body, an intake opening through the **piston**, a discharge opening through the end plate, a first porous frit seated within a frit-receiving socket of the **piston** and covering the intake opening, and a second porous frit secured to the end plate and covering the discharge opening. A slurry containing the packing material, such as a granular silica or polymeric media, is placed within the column body and the **piston** is moved toward the fixed end plate to compress the slurry. The pores of the frits are sized to permit the liquid of the slurry to flow out the discharge opening while preventing discharge of the packing material. Conventionally, when the chromatographic media within a chromatographic column is packed, a telescoping rod of a hydraulic pushing device pushes the **piston** into the column. This compression packs the packing material to a predetermined pressure (which may

typically be around 1,000 to 5,000 p.s.i., but these values are merely exemplary rather than limiting.

Brief Summary Text - BSTX:

With such conventional method of packing chromatographic columns, the column remains attached to the pushing device so that the rod of the hydraulic pushing device maintains the pushing force on the **piston**. In other words, the column must remain attached to the pushing device during operation of the chromatographic column, i.e., during chromatographic separations.

Brief Summary Text - BSTX:

Among the several objects of the present invention may be noted the provision of an improved method and apparatus for packing chromatographic columns; the provision of such a method and apparatus for packing a chromatographic column in which compression is maintained on a column **piston** even after removal of the column from a pushing device; the provision of such a method and apparatus in which the column is permitted to move in an axial direction to compress a chromatographic medium in the column but is prevented from moving in an opposite direction; and the provision of such a method and apparatus in which an elastic force is applied to the **piston** even after removal of the chromatographic column from the pushing device.

Brief Summary Text - BSTX:

In general, a chromatographic device of the present invention comprises a longitudinally extending tube having a first end and a second end, the second

end being closed, a **piston** inside the hollow tube, a chromatographic medium, a rod, and a locking mechanism. The chromatographic medium is within the tube and between the **piston** and the second end. The rod has a longitudinal rod axis and is operatively engageable with the **piston** for pressing the **piston** in the hollow tube. The locking mechanism is adapted for locking the rod relative to the tube in a manner to prevent movement of the rod relative to the tube in an outward longitudinal direction away from the **piston** without preventing movement of the rod relative to the tube in an inward longitudinal direction toward the **piston**.

Brief Summary Text - BSTX:

Another aspect of the present invention is a method of packing a chromatographic medium in a longitudinally extending chromatographic tube. The tube has a first end and a second end, the second end being opposite the first end and closed. The method comprises: placing the chromatographic medium in the chromatographic tube; inserting a **piston** into the tube, the chromatographic medium being between the **piston** and the closed second end of the tube; placing a pushing assembly adjacent the **piston**, the pushing assembly having a locking mechanism adapted for permitting the **piston** to move in a first longitudinal direction toward the closed second end of the tube and for preventing the **piston** to move in a second longitudinal direction toward the first end of the tube; applying a compressive force in the first longitudinal direction against the pushing assembly to move the **piston** toward the closed second end of the tube to compress the chromatographic medium; and removing the compressive force

from the pushing assembly, the locking mechanism locking against movement of
the **piston** in the second longitudinal direction.

Drawing Description Text - DPTX:

FIG. 1 is a front elevational view of a chromatographic device of the present
invention in partial section, the chromatographic device including a column
body, a chromatographic medium, a **piston**, and a pushing assembly;

Drawing Description Text - DPTX:

FIG. 3 is a fragmented front elevational view, in section, showing a locking
mechanism of the pushing assembly positioned to prevent movement of a rod of
the locking mechanism away from the **piston**;

Drawing Description Text - DPTX:

FIG. 4 is a fragmented front elevational view, in section, similar to that of
FIG. 3 but showing the position of the locking mechanism as the rod is being
pushed toward the **piston**;

Drawing Description Text - DPTX:

FIG. 5 is a fragmented front elevational view, in section, similar to that of
FIGS. 3 and 4, but showing a release member positioned to permit the rod to
move away from the **piston**;

Detailed Description Text - DETX:

Referring now to the drawings, and first more particularly to FIG. 1, a
chromatographic device of the present invention is indicated in its entirety by

the reference numeral 20. The chromatographic device includes a tubular column body, generally indicated at 22, a chromatographic medium 24, a **piston** 26, and a pushing assembly, generally indicated at 28. Preferably, the column body 22, chromatographic medium 24, and **piston** 26 are substantially the same as those described in U.S. patent application Ser. No. 08/942,153, filed Oct. 1, 1997 and entitled Apparatus That Maintains Compression in a Tube, incorporated herein by reference.

Detailed Description Text - DETX:

The column body 22 comprises a longitudinally extending tube 30 having a first end 32 (the upper end as viewed in FIG. 1) and a second end 34 (the lower end as viewed in FIG. 1) opposite the first end. A first (upper) flange 36 circumscribes the tube 30 at the first end 32, and a second (lower) flange 38 circumscribes the tube at the second end 34. The upper and lower flanges 36, 38 preferably are rectangular, and more preferably are square. An end plate 40 closes the lower end 34 of the tube 30 and is secured to the lower flange 38. The end plate 40 is shaped and configured for holding one or more porous frits 42, and includes a plurality of radial grooves 44 and a discharge opening for receiving an outlet tube 46. The **piston** 26 is moveable within the tube 30 of the column body 22. The **piston** 26 also includes frits (not shown, similar to frits 42. The frits allow passage of a fluid containing substances in solution to be separated while not allowing solid particles in the chromatographic medium (usually, but not necessarily, fine silica particles) to escape from the column body 22. The solution is supplied through a feeding tube 48 secured to the **piston** 26 and in fluid communication with a passageway

(not shown)
extending through the **piston**. It is to be understood that the designation of
one of the tubes as a "feeding" tube and the other as an "outlet" tube is
arbitrary, inasmuch as the chromatographic device 20 is capable of being
operated with fluid flowing through it in either direction.

Detailed Description Text - DETX:

Referring now to FIGS. 1-5, the pushing assembly 26 comprises a spring
assembly, generally indicated at 50, a rod 52, and a locking mechanism,
generally indicated at 54. As described in greater detail below, the pushing
assembly 26 is configured to be positioned against the **piston** 36 in a manner
such that application of a compressive force (i.e., a downward force as shown
in FIG. 1) on the rod 52 moves the **piston** downward to pack the chromatographic
medium 34.

Detailed Description Text - DETX:

The locking mechanism 54 includes a base member 64, a cover plate 66, a
belville spring 68, four rectangular locking plates (or members) 70, and a
canting member 72. The locking mechanism 54 is configured for locking the rod
52 relative to the base member 64 in a manner to prevent movement of the rod
relative to the base member in an outward longitudinal direction (up as viewed
in FIG. 1) away from the **piston** without preventing movement of the rod relative
to the base member in an inward longitudinal direction (down as viewed in FIG.
1) toward the **piston**. The base member 64 includes a bore 74 sized for
receiving the belville spring 68 and the canting member 72, and a cavity 76
above the bore 74 for receiving the locking plates 70 in a

face-to-face
orientation. The base member 64 is releasably secured to the upper flange 36
of the column body 22 via four pin-type fasteners 78 (only two of which are
shown). Preferably, the pin-type fasteners 78 are of the type manufactured by
Vlier Inc., 40 Guest Street, Brighton, Mass., under the description full travel
hand-retractable plungers. The fasteners 78 include retractable pins (not
shown) adapted to be laterally inserted into horizontal bores (not shown) in
the upper flange 36.

Detailed Description Text - DETX:

In operation, the column body 22 is placed on a pushing device, such as the
compression apparatus 86 (FIGS. 6 and 7), with the upper flange 36 of the
column body supported on a column receiving portion 85 of the compression
apparatus. Chromatographic medium 24 is then poured or otherwise placed into
the tube 30 of the column body 22 via the upper end 32 of the tube. Next, the
piston 26 is inserted into the tube 30 and pushed downward toward the medium
24. The pushing assembly 28 is then positioned with the spring assembly 50
inserted into the tube 30 of the column body 22 and the base member 64 placed
over the upper end 32 of the tube and secured to the upper flange 36.
Preferably, the spring assembly 50 includes upper and lower disc-shaped guide
blocks 86, 88. The upper guide block 86 is secured to the upper spring
engaging member 56 and the lower guide block 88 is secured to the lower spring
engaging member 54. The guide blocks 86, 88 keep the spring engaging member 56
centered within the tube. The guide blocks 86, 88 are configured to slide
within the tube 30 and are preferably made of polytetrafluoroethylene.

Preferably, the guide blocks 56, 58 include slots for passage of the feeding tube 48 therethrough. The underside of the lower spring engaging member 34 is brought into engagement with the upperside of the **piston** 26. The rod 52 of the pushing assembly 28 extends upward through the locking mechanism 54 and its longitudinal axis X.sub.1 is coaxial with the axis of the column body 22.

Detailed Description Text - DETX:

The compression apparatus 84 includes a hydraulic cylinder 90 having a pushing rod 92. The pushing rod 92 has a longitudinal axis which is aligned with the axis X.sub.1 of the rod 52 of the pushing assembly 28. The pushing rod 92 is hydraulically driven along the axis X.sub.1 between raised and lowered positions. The pushing rod 92 is driven downward against the upper end of the rod 52 of the pushing assembly 28 to press the spring assembly against the **piston** 26 to thereby **drive the piston** downward against and pack the chromatographic medium 24. The locking mechanism 54 permits downward (inward) movement of the rod 52 relative to the column body 22. After the chromatographic medium 24 is sufficiently packed, the pushing rod 92 is driven upward away from the rod 52 of the pushing assembly 28. The locking mechanism 54 prevents upward movement of the rod 52 relative to the column body 22 and relative to the base member 64. The downward movement of the rod 52 through the column body 22 caused the spring 60 to compress and thereby exert an elastic force on the **piston** 26 to press the **piston** against the chromatographic medium. Because the rod 52 is locked against upward movement relative to the column body 22, the spring 60 remains compressed even after the pushing rod 92

of the compression apparatus 84 is raised. Thus, the spring 60 of the pushing assembly 28 maintains a pushing force (i.e., constant axial compression) against the **piston** 26 to prevent the formation of voids in the packing medium 24. After the pushing rod 92 is raised, the assembled chromatographic device 21 may be removed from the compression apparatus 84 and then used for a chromatographic separation operation at a location remote from the compression apparatus.

Claims Text - CLTX:

inserting a **piston** into the tube, the chromatographic medium being between the **piston** and the closed second end of the tube;

Claims Text - CLTX:

placing a pushing assembly adjacent the **piston**, the pushing assembly having a spring, a rod, and a locking mechanism adapted for permitting the **piston** to move in a first longitudinal direction toward the closed second end of the tube and for preventing the **piston** to move in a second longitudinal direction toward the first end of the tube;

Claims Text - CLTX:

applying a compressive force in the first longitudinal direction against the pushing assembly to move the **piston** toward the closed second end of the tube to compress the chromatographic medium;

Claims Text - CLTX:

removing the compressive force from the pushing assembly, the locking mechanism

locking against movement of the **piston** in the second longitudinal direction.

Claims Text - CLTX:

2. A method as set forth in claim 1 wherein the pushing assembly includes the locking mechanism, a rod, and a spring, wherein the step of placing a pushing assembly adjacent the **piston** comprises positioning the pushing assembly so that the spring is positioned between the rod and the **piston**, and wherein the step of applying a compressive force comprises applying a compressive force against the rod to compress the spring so that the spring transmits the compressive force to the **piston**.

Current US Original Classification - CCOR:

210/656

Current US Cross Reference Classification - CCXR:

210/198.2

US-PAT-NO: 5919361
DOCUMENT-IDENTIFIER: US 5919361 A

TITLE: Spring-loaded hydraulically active liquid chromatography column

DATE-ISSUED: July 6, 1999

US-CL-CURRENT: 210/198.2; 210/656

APPL-NO: 08/ 992055

DATE FILED: December 17, 1997

---------- KWIC ---------

Abstract Text - ABTX:

The adsorbent bed (25) retained in an HPLC column (1) is maintained in a constant state of compression by a spring loaded hydraulically active second column (13). A relatively small spring (14) loaded **piston** (15) in the second column maintains a constant hydraulic pressure in the hydraulic chamber (9) of the larger diameter HPLC column (1). Multiple small diameter columns (13) can be employed if necessary.

Brief Summary Text - BSTX:

U.S. Pat. No. 3,169,522 issued to Y. Shalon et al. describes an hydraulically active liquid chromatography column suitable for use in HPLC systems. A cylindrical bed of adsorbent is retained within an adsorbent chamber and is compressed by a **piston** driven by a hydraulic fluid in the hydraulic chamber. An opening is provided in the end plate of the apparatus for enabling the

monitoring, maintaining and releasing of the pressure in the column as necessary. This opening may be equipped with a gauge and valve.

Brief Summary Text - BSTX:

FIG. 2 of an article by Yehuda Shalon printed in the magazine American Laboratory in September 1997 illustrates a spring-loaded HPLC column. The **piston** applying pressure to the cylindrical bed of adsorbent may be driven downward through a threaded screw at an upper end of the column. A compressed spring intermediate the **piston** and the threaded screw acts directly on the **piston**. The spring maintains a relatively constant pressure upon the bed of adsorbent.

Brief Summary Text - BSTX:

One broad embodiment of the invention may be characterized as an apparatus for performing an adsorptive separation comprising a sealed first column having a cylindrical internal volume and closed first and second ends; a bed of adsorbent located within an adsorbent chamber located within the first column, with the bed of adsorbent being compressed by a movable **piston** located in the internal volume of the first column and driven by hydraulic pressure maintained in a first hydraulic chamber located in the internal volume of the first column between the **piston** and the first end of the first column; process fluid transfer lines for delivering and removing a process fluid, which transfer lines are connected to opposite ends of the adsorbent chamber; a sealed second column having first and second ends, with a second movable **piston** being located within the second column and a spring being located within

the second column
between the first end and the second movable **piston,** with the spring arranged
to force the second **piston** toward a second hydraulic chamber which is located
between the **piston** and the second end of the second column; and, a hydraulic
fluid transfer connection between the first and second hydraulic chambers.

Detailed Description Text - DETX:

Hydraulically active columns apply pressure sufficient to axially compress the
adsorbent bed. However, the applied pressure is not regulated in a manner
which adjusts for the changing pressures imposed during an SMB cycle. To
overcome this problem the art has developed the use of spring loaded columns in
which a compressed spring is used to apply a pressure to the adsorbent bed. As
the spring can expand and contract, the bed can also expand and contract
somewhat due to pressure changes. However, spring loaded columns are limited
by the practical size of the spring which can be used. When it is desired to
scale up the separation process to an intermediate scale in which the adsorbent
chamber has a diameter of 25 cm or more the total force on the **piston** used to
compress the cylindrical bed of adsorbent can be very large and it is
impractical to employ a spring. For instance the pressure on a 75 cm diameter
piston having an operational pressure of 1,500 psig is about 1,000,000
lbs.sub.f. Simply put the required spring size is impractical for an HPLC
column.

Detailed Description Text - DETX:

Referring now to FIG. 1, the largest component of the apparatus is the primary

cylinder or column 1 which functions as a hydraulically-loaded (compressed) chromatography column. During use and operation the primary column 1 is sealed at its upper end by a top flange 11 and at its lower end by a lower flange 22. This forms a cylindrical internal volume which is divided into two chambers by a movable first **piston** 4. One or more O-rings 6 and/or seal rings 7 can be present on the **piston** to help prevent passage of either fluid in the column around the **piston** 4. The volume below the **piston** in this view, with this direction being relative to the normal flow of liquid in the column, comprises a cylindrical adsorbent chamber 2. During operation the chamber 2 is filled with a densely packed bed of solid particulate adsorbent. The remainder of the internal volume of the primary column 1 is comprised of the hydraulic chamber . This chamber 3 fills the space between the flange 11 and the **piston** 4 at the upper end of the apparatus.

Detailed Description Text - DETX:

During operation a process stream is charged to the column through process line 10. During a complete SMB cycle, the composition and pressure of the process stream will vary greatly. The transfer lines are connected to the apparatus through fittings of customary design threaded into the flanges. The incoming process stream is thereby delivered to a flexible transfer line 5 present in the hydraulic chamber 3. The transfer line 5 is shown as coiled not to indicate that it is spring-like but rather to indicate its flexible nature which will allow the transfer line to accommodate any movement of **piston** 4. The **piston** could travel to the lower end of the primary column when for instance an adsorbent bed is being replaced with different

or fresh adsorbent.
At this time the lower flange 22 is removed and the **piston** is used to simply
push the adsorbent out of the bottom of the column.
However, during normal
operation the **piston** is essentially fixed in position.

Detailed Description Text - DETX:

The incoming process stream of line 20 is thereby delivered by line 8 to the
transfer channel 24 extending through the **piston**. The channel delivers the
liquid to the fluid distribution frit 5 on the face of the **piston**. A liquid
distribution means not shown may be located between the face of the **piston** and
the frit for purposes of ensuring uniform distribution of the incoming liquid
across the upper surface of the adsorbent bed. This is important as it is
desired to maintain plugflow conditions across the cross section of the
adsorbent bed. At the outlet end of the adsorbent chamber, a related problem
occurs in that there should be uniform collection of the liquid across the
entire cross section of the adsorbent bed. There is therefore provided a
liquid collection and frit means 23 having a number of channels or other open
volumes which allow free flow of the liquid to a central conduit leading out of
the chamber. The process stream then flows through a port in the outlet flange
22 through a threaded fitting and into the outlet transfer line 21. The
transfer line 21 and the transfer line 20 may be connected to valves regulating
the flow of the process liquid or directly to the next downstream adsorbent
chamber or directly to storage tanks for the feed and product streams.

Detailed Description Text - DETX:

The third fluid transfer port 9 in the inlet flange 11 is directly connected via a screw-on coupling to a smaller second column 13. The secondary column 13 is preferably also cylindrical but has a much smaller diameter and cross-sectional area than the primary column 1. A flange 16 is shown as closing only the left-hand end of the column 13 but flanges could be used at both ends of the column if desired. The secondary column 13 differs from the primary column 2 in that it does not contain any adsorbent. Instead the secondary column has a hydraulic chamber 19 which is filled with the same hydraulic fluid as the hydraulic chamber 3 of the primary column 1. The open fluid communication between these two hydraulic chambers through the port 9 causes essentially instantaneous equalization of the pressures in the two chambers. The secondary column 13 has a spring 14 which drives a piston 15 against the hydraulic chamber of the secondary column. By proper choice of the size and strength of the spring 14, any change in the operating pressure in the adsorbent chamber of the primary column can be easily compensated for. Any settling or other disturbance of the adsorbent bed in the adsorbent chamber 2 of the primary column will result in a corresponding movement of the piston 4 in the primary column. However, this movement will not reduce the pressure in the hydraulic cylinder 3 and thus will not reduce the pressure applied onto the adsorbent bed because of the action of the spring 14 against the secondary hydraulic chamber 19.

Detailed Description Text - DETX:

FIG. 2 illustrates the use of a plurality of secondary columns 13 to maintain a desired pressure in a primary hydraulic chamber 3 of a much

larger adsorption
column 1. For instance, if this drawing was to scale and each of the smaller
secondary columns 13 is approximately 5 centimeters in diameter then the larger
adsorption column 1 would be about 20 centimeters in diameter. A plurality of
smaller columns may be desired when a very high ratio in column cross section
exists. Movement of the large **piston** a short distance may require the delivery
or removal of a large percentage of the volume of a single small column. The
ratio in **piston** diameters and corresponding cross-sectional area of the two
columns can be much higher than this. A ratio of ten to one or more in **piston**
diameters is possible. The total force imposed upon the larger **piston** 4 by the
pressurized fluid can therefore be extremely high due to the large surface area
of the **piston**. Attempting to compensate for changes in such a large load
through the use of a single or even multiple springs attached to the **piston** 4,
would result in the need for excessively large and powerful springs. Instead
the invention employs the hydraulic force supplied by the three smaller
secondary chambers 13 each having a spring 14 which pushes upon a small **piston**
15. This **drives** hydraulic fluid from the chamber 19 through the connecting
lines 26, 27, 28, 29 and 30 into the hydraulic chamber 3 of the primary column
of the apparatus. The hydraulic fluid may also flow from the primary hydraulic
chamber to the secondary chambers. As the force applied against the **pistons** 15
in the secondary chambers 13 is much smaller due to the smaller surface area of
the **pistons,** much smaller and more practically sized springs 14 are sufficient.
Also shown on this drawing, is a valve 31 in the fluid transfer line 18 for the
hydraulic fluid to and from the hydraulic chamber 3 of the primary adsorption

column 1. Another feature shown on this Figure is the adsorbent bed 25 located between the **piston** 4 and the outlet end of the adsorbent chamber.

Claims Text - CLTX:

b. a bed of adsorbent located within an adsorbent chamber located within the first column, with the bed of adsorbent being compressed by a movable **piston** located in the internal volume of the first column and driven by hydraulic pressure maintained in a first hydraulic chamber located in the internal volume of the first column between the **piston** and the first end of the first column;

Claims Text - CLTX:

d. a sealed second column having first and second ends, with a second movable **piston** being located within the second column and a spring being located within the second column between the first end and the second movable **piston,** with the spring arranged to force the second **piston** toward a second hydraulic chamber which is located between the **piston** and the second end of the second column; and,

Current US Original Classification - CCOR:

210/198.2

Current US Cross Reference Classification - CCXR:

210/656

US-PAT-NO: 5667675
DOCUMENT-IDENTIFIER: US 5667675 A

TITLE: Collapsible apparatus for compressing packing material in liquid chromatography columns and methods of use

DATE-ISSUED: September 16, 1997

US-CL-CURRENT: 210/198.2; 210/241 ; 210/656 ; 96/101

APPL-NO: 08/ 609967

DATE FILED: February 29, 1996

---------- KWIC ---------

Abstract Text - ABTX:

Longitudinally collapsible apparatus for compacting packing material within a tubular liquid chromatography column. The apparatus includes a first support member, a second support member and a movable support member. The movable support member is releasably connectable to the second support member for support thereby. When released from the second support member, the movable member is adapted to ride on guide members extending between the first and second support members. A compression mechanism is supported on the movable support member to move therewith to a longitudinally collapsed position for the apparatus. The compression mechanism comprises a reversible **drive** for an axially movable rod carrying a movable **piston**. With the movable support member secured to the second support member, the **piston** of the compression mechanism is movable in an end of the tubular column when the column is supported on the

first support member. Movement of the movable **piston** within the column axially compresses a column packing material as the **piston** is driven towards an opposite end of the column. The **piston** also is releasably connectable to the tubular column to raise and lower the column when the apparatus extends in a vertical direction and if it is desired to insert an end **piston** into or remove the end **piston** from the opposite end of the column or when it is desired to change an adapter ring on the first support member to receive a column of a different diameter for packing by the apparatus.

Brief Summary Text - BSTX:

Most preparative chromatography is performed in tubular metal columns of 2, 4 or 6 inch or larger diameter. Preparation of such large diameter columns is difficult using conventional slurry packing wherein a suspension of stationary phase is forced or "packed" into a chromatographic column using a high pressure stream of liquid to produce a homogeneous and stable column structure. Rather, today, the preferred method of preparing large diameter columns for use in preparative chromatography is the method commonly referred to as "axial compression". Axial compression is described in U.S. Pat. Nos. 3,966,609 and 5,169,522. In the '609 patent a **piston** containing a filter matrix and fluid sealing mechanism is used to compact a bed of stationary phase and maintain adequate compression to correct column instability. In the '522 patent a compression ram is used to consolidate the stationary phase during the packing operation.

Brief Summary Text - BSTX:

The present invention provides apparatus for producing and operating highly efficient preparative columns while avoiding the disadvantages of the prior art. In the present invention, a tubular column or column tube is provided with end closures containing filters capable of retaining the stationary phase and provisions for the introduction and removal of liquid under pressure. The end closures, generally known as bed supports, are supported within opposite ends of the column tube against outward axial motion by **piston** elements which provide both fluid connections for the passage of mobile phase and high pressure sealing elements to prevent leakage of the mobile phase from the column. At least one of the bed supports and its supporting **piston** are movable within the column tube and are subjected by means of a compression mechanism to a compressive force larger than that generated by the pressure drop occurring from the passage of liquid through the stationary phase. The compressive force permits the preparation and continued operation of high performance liquid chromatographic columns.

Brief Summary Text - BSTX:

In a typical embodiment of the invention, the compressive mechanism comprises a hydraulic press including a vertically extending hydraulic cylinder containing a compression rod supporting a compression **piston** at its lower end. The compression **piston** functions as the upper support **piston** for the column tube containing packing material to be compressed by the apparatus of the present invention. The cylinder is mounted on a movable support member which is mounted for vertical movement on vertical guide members extending between upper and lower support members. The lower support member is

designed to support the column tube containing packing material which is to be compressed. The movable support member is designed for releasable connection to the upper support member. When the movable member is released from the upper support member it is movable downward to vertically collapse the apparatus of the present invention. When the movable support member is connected to the upper support member, the compression **piston** is movable axially into an upper open end of the column tube which is vertically mounted on the lower support member. Upon actuation of the compression mechanism, the compression rod is driven downward to **drive** the compression **piston** into the column tube to engage an upper bed support and to compact the packing material within the column tube against a lower bed support and a stationary lower **piston** within a lower end of the column tube. The compression **piston** and the lower stationary **piston** include annular fluid seals for sealing with an inner surface of the column tube and contain fluid passages for the introduction and removal of mobile phase. The lower support member is provided with an opening through which spent packing may be extruded. This opening is reduced in diameter for packing and operation by the mounting of an adapter plate or ring. The adapter ring may be selected from a variety of diameters to permit the operation of columns of different diameters using the same compression mechanism.

Brief Summary Text - BSTX:

To prepare a liquid chromatography column using the apparatus of the present invention, the correct size adapter ring is mounted on the lower support member. The lower **piston** and bed support are then mounted on the lower support

member via the adapter ring, and the column tube is pressed downward onto the
lower bed support and lower **piston** to close the lower end of the column tube.
A suspension of stationary phase is introduced into the upper end column tube,
which is then closed by the upper bed support. At least one of the bed
support-**piston** sets is provided with a connection for liquid to exit the
apparatus during processing. The upper movable bed support is then subjected
to a compressive force by the compression **piston,** which causes the movable bed
support to travel downward within the column tube in an axial direction until
excess slurry solvent is expelled and the stationary phase is compacted. The
column is then ready for operation and may be connected to a liquid
chromatographic system for use. There is no need for additional processing
operations and the column is suitable for additional compression, unlike those
prepared in accordance with U.S. Pat. No. 4,549,584.

Brief Summary Text - BSTX:

In the present invention, the stationary phase in the column is operated under
compression by the movable upper **piston**. However, both the upper and lower
pistons can be removed from the column tube without removing the bed supports.
Since there is no need to remove the movable bed supports from the column tube,
there is reduced danger of damage to the stationary phase structure from
removal of the **pistons**. The compression mechanism and **pistons** can then be used
to prepare additional columns without destroying the columns previously
prepared using the compression mechanism. Also, columns previously prepared
using the compression mechanism may be recompressed and again operated under
compression to maintain column performance.

Brief Summary Text - BSTX:

It is a further feature of the present invention that the use of interchangeable adapter plates or rings and **piston** sets permits the use of the compression mechanism with columns of differing diameters without encumbering each column with complex and costly flanges or other devices to connect to fluid flow passages. This allows column tubes according to the present invention to be of simple design and constitutes an advantage over previous designs.

Brief Summary Text - BSTX:

After the use of chromatographic column of the present invention is complete, the spent stationary phase is easily removed from the column by raising the column tube, removing the lower **piston,** lowering the column tube and using the compression mechanism to extrude the stationary phase through the opening in the lower support member. The bed supports are easily removed by the same extrusion. The use of the adapter plates and lower **piston** provides for this simple extrusion process, which is not present in the Merck system.

Brief Summary Text - BSTX:

In the present invention, the hydraulic press is adapted to two further uses beyond the compression of the stationary phase for operation or extrusion. First, the hydraulic press is used to raise and lower the column tube during the operations of inserting the lower bed support and **piston,** and during the removal of the **piston** before stationary phase extrusion.

Since large diameter column tubes can weigh more than 50 pounds even when empty, such use of the hydraulic press provides a significant advantage to the operator. The operator need only mount the column tube on the lower support of the apparatus. All subsequent lifting and lowering is performed by the apparatus itself. In particular, such column raising and lowering is accomplished by coupling the upper end of the column tube so that the tube follows the motion of the compression rod. The coupling can be performed using a number of methods. The preferred method is by using a removable pin which passes through a hole through the column tube and the **piston** at a right angle to the tube axis.

Drawing Description Text - DRTX:

FIG. 1a is a an exploded view of a column packing compression mechanism shown in FIG. 1 illustrating the component part comprising a vertically extending compression rod, a **piston** at a lower end of the compression rod and a **drive** for vertically moving the compression rod comprising a hydraulic cylinder.

Drawing Description Text - DRTX:

FIG. 1b is a side view showing the components in FIG. 1a assembled with the **piston** secured to the compression rod by a universal coupler and a laterally extending pin, the **piston** shown in FIGS. 1, 1a and 1b being a "dummy" **piston** for use when the apparatus of the present invention is to be vertically collapsed.

Drawing Description Text - DRTX:

FIG. 2 is a side view partially in section of the apparatus of FIG. 1 with the moveable support plate secured to an upper support member and with the moveable support plate supporting the hydraulic cylinder in an upward extended position with a compression rod of the column packing compression mechanism and "dummy" **piston** bearing on the lower support member.

Drawing Description Text - DRTX:

FIG. 3 is a side view partially in section showing an adaptor ring mounted on a lower support member of the apparatus and vertically supporting a column tube between the lower support member and the moveable and upper support members. The compression rod of the column packing compression mechanism is shown supporting a compression **piston** for fitting into an upper open end of the column tube, FIG. 3 also depicts a front face of the control panel for controlling operation of the apparatus.

Drawing Description Text - DRTX:

FIG. 4 is a side view partially in section similar to FIG. 3 and illustrating the compression rod and compression **piston** releaseably secured to the column tube by a pin extending through aligned openings in the column tube and compression rod above the compression **piston**. FIG. 4 shows the compression rod in a raised position to lift the column tube from the adaptor ring and lower support member, the adaptor ring shown supporting a lower **piston** and bed support for closing the lower open end of the column tube. The lower **piston** includes openings for passing liquid from the column tube during operation of the column.

Drawing Description Text - DFTX:

FIG. 5 is a side view partially in section of the apparatus of FIG. 4 showing the column tube after it has been driven downward onto the adaptor ring to receive the lower **piston** and lower end closure.

Drawing Description Text - DFTX:

FIG. 6 is a side view partially in section of the apparatus of FIG. 5 after the connecting pin has been removed to release the compression **piston** and compression rod from the column tube, the compression rod and compression **piston** being depicted in a raised position with the upper end of the column tube open to receive a slurry of the packing material for packing upon a downward movement of the compression **piston** into the column tube.

Detailed Description Text - DETX:

The apparatus 10 further comprises a column packing compression mechanism 22 comprising a compression rod 24, a compression **piston** 26 and a **drive** 28 preferably including a hydraulic cylinder 30 having lower and upper flanges 32 and 34 respectively secured by tie rods 36. The column packing compression mechanism 22 is secured to movable support member 18 for vertical movement therewith to vertically collapse 10 when the moveable support member 18 is released from the upper support member 12 as depicted in FIG. 1.

Detailed Description Text - DETX:

When the moveable support member 18 is secured to the upper support member 12,

operation of the **drive** 28 by actuation of knobs on a control panel 37 cause the compression rod 24 to move vertically to raise and lower the compression **piston** 26 which is sized to fit snugly into an open upper end of a column tube 35 as shown in FIGS. 3, 4, 5 and 6. The column tube is supported on the lower support member 14 over the central hole 15 and downward movement of the compression **piston** 26 within the column tube compresses a packing material within a lower end of the column tube.

Detailed Description Text - DETX:

As previously indicated, the upper support member 12 is adapted to secure the moveable support member 18 thereto when it is desired to operate the apparatus 11 in compressing a packing material within the column tube 35. To accommodate such connection, the support blocks 64 and 66 include through holes 64a and 66a respectively. Such holes are designed to receive vertically extending bolts 64b and 66b passing through the support blocks 64 and 66 into threaded holes 69 moveable support member 18 (see FIG. 1c) to releaseably secure the moveable support member to the upper support member 12 as shown in FIG. 2a. Thus secured to the upper support member 12, the moveable support member 18 provides vertical support for the hydraulic cylinder 30 of the column packing compression mechanism 22 above the upper support member 12 as shown in FIG. 3. In this regard, and as most clearly shown in FIGS. 1c and 1d, the moveable support member 18 comprises a flat plate including a central stepped opening 71 defining an annular shoulder 72 for receiving a lower face of a cylinder gland 73 of the hydraulic cylinder 30. Thus positioned, the lower flange 32 of the cylinder rests on the top face of the moveable support

plate 18 with bolts
extending through mating threaded holes 74 in the moveable support plate and
lower flange 32. The threaded holes 74 receive bolts 75 to releaseably secure
the flange to the moveable support plate as shown most clearly in FIG. 1. With
the column packing compression mechanism 22 thus supported on the moveable
support member 18 as shown in FIG. 1, the compression rod 24 extends axially
from the cylinder 30 through the opening 71 in the moveable support plate 18 to
releaseably secure the compression **piston** 26 (FIG. 3) in line with an open
upper end of the column tube 35 mounted on the lower support member 14 as shown
in FIG. 3. Such connection of the compression rod 24 to the compression **piston**
26 is most clearly depicted in FIG. 3 as well as in FIGS. 1a and 1b for a
"dummy" **piston** connected to the compression rod 24. As there illustrated, the
lower end of the compression rod 24 includes a threaded socket 77 for receiving
an externally threaded upper end of a cylindrical coupler 78 to secure the
coupler to the compression rod 24. The coupler 78 also include a lower socket
80 having laterally extending side holes there through. The socket 80 is
dimensioned to receive the upper end of the compression **piston** 26 in FIG. 3 or
a "dummy" **piston** 26a as depicted in FIGS. 1a and 1b. In both instances, the
piston includes a laterally extending through hole for receiving a connecting
pin 82 after the **piston** has been inserted upwardly into the lower socket 80 as
most clearly depicted in FIGS. 1b and 3.

Detailed Description Text - DETX:

In the vertically collapsed position for the apparatus 10, the "dummy" **piston**
26a bears adaptor ring 46 mounted on the lower support member 14. In the

raised position for the moveable support member 18 as depicted in FIGS. 3-6, the compression system 22 is raised from the lower support member 14 which provides vertical support for the column tube 35 containing packing material which is to be compressed upon downward movement of the compression **piston** 26 into the column tube. To provide such support for the "dummy" **piston** 26a as shown FIGS. 1 and 2 and for the column tube 35 as shown in FIGS. 3-6, the lower support plate 14 includes the previously described adaptor ring 46. As shown most clearly in FIG. 1f, the adaptor ring includes an annular recess 84 forming an annular shoulder 85 for receiving a bottom face of the "dummy" **piston** 26a as depicted in FIGS. 1 and 2 or the lower end of the column tube 35 as depicted in FIGS. 3-6.

Detailed Description Text - DETX:

As previously indicated, user control of the controls on the control panel 37 determines the operation of the apparatus 10 in moving to and from the vertically collapsed position shown in FIG. 1 and in the placement of the column tube 35 and its end closures and **pistons** in the ends of the column tube and the subsequent compression of a packing material within the column. To accomplish this, the column packing compression mechanism 22 as previously described preferably is a model 2H hydraulic ram manufactured by Parker-Hannifin Corp. of Des Plaines, Ill. The hydraulic ram is connected in a conventional manner and controlled by the fluid circuit depicted in FIG. 7 under control of the controls indicated on the control panel 37. In particular, the ram comprises the hydraulic cylinder 30 with the compression rod 24 extending downwardly therefrom. Fluid to control

the raising and lowering of the compression rod within the cylinder is controlled by operation of a directional control valve 90 shown in FIG. 7 and on the control panel 37 in FIGS. 1, 2 and 3 through 6.

Detailed Description Text - DETX:

The directional valve 90 is a conventional three position valve for regulating the direction of fluid flow to and from the hydraulic cylinder 30. The hydraulic cylinder includes an internal **piston** for driving the compression rod 24 in and out of the hydraulic cylinder in a conventional manner. For example, with the directional control valve 90 in the position indicated, fluid flows into an upper end of the hydraulic cylinder 30 and returns from a lower end of the cylinder to **drive** the compression rod 24 downwardly and out of the hydraulic cylinder. When the directional control valve 90 is in its second or intermediate position, all flow of fluid to the hydraulic cylinder 30 is blocked. When the control valve is in its lower or third position, the direction of flow is reversed such that fluid flows into the lower end of the hydraulic cylinder 30 below its internal **piston** and exits the hydraulic cylinder at the upper end for return to the reservoir 92.

Detailed Description Text - DETX:

In this regard, and as previously indicated, the apparatus 1 is susceptible as several different operations. First, as depicted in FIG. 1 and 2, the apparatus may be vertically collapsed to reduce the vertical dimension of the apparatus to a minimum allowing for compact packaging and shipping of the apparatus as well as for movement of the apparatus from one

location to another
in a laboratory through doorways and the like without
requiring a tilting or
rotation of components of the apparatus. Such collapsing
of the apparatus 10
is accomplished by removing the column tube 35 from the
apparatus and with the
"dummy" **piston** 26a attached to the lower end of the
compression rod 24, setting
the control knob "RAM" to the "lower" position. This
results in the
compression rod being driven downwardly to the position
indicated in FIG. 2
with the "dummy" **piston** bearing on the top of the adaptor
ring 46 secured to
the lower support member 14. Then, upon a disconnection of
the connector 20
securing the moveable support member 18 to the upper
support member 12,
continued operation of the hydraulic system with the RAM
control in the "raise"
position lowers the hydraulic cylinder 30 with the moveable
support member 18
downwardly on the guide rods 16 to the lower or collapsed
position shown in
FIG. 1.

Detailed Description Text - DETX:

When it is desired to again extend the apparatus 10, the
process is simply
reversed. With the hydraulic cylinder 30 in the lower
position shown in FIG.
1, the control knob "RAM" is switched to the "lower"
position. This **drives** the
hydraulic cylinder 30 and moveable support member 18
upwardly to the position
shown in FIG. 2 where the connector 20 again releaseably
locks the moveable
support member to the upper support member. Continued
operation of the
hydraulic system then with the control knob "RAM" in the
"raise" position
withdraws the compression rod 24 upwardly within the
hydraulic cylinder 30 to
the position shown in FIG. 3 where the "dummy" **piston** 26a
may be replaced by

the compression **piston** 26 and the apparatus 10 readied to receive a column tube
35 as shown in FIG. 3.

Detailed Description Text - DETX:

As shown in FIG. 3, the adaptor ring 46 of proper internal diameter is first
secured to the top of the lower support plate 14 and an empty column tube 35
mounted thereon as shown. As depicted in FIG. 4, the compression rod and
piston are then driven downwardly into an upper end of the empty column tube.
Next, the compression rod and **piston** are releaseably secured to the column tube
by a connector pin 98 passing through aligned holes 99 in the column tube and
the **piston** coupler 76 as shown in FIG. 4. Upward movement of the compression
rod and **piston** then lifts the column tube 35 from the adaptor 46 allowing a
lower **piston** and bed support 100 including filters and fluid ports to be
located on the adaptor ring as illustrated. Next, the compression rod and
piston are lowered to lower the column tube 35 onto the bed support and lower
piston 100 as depicted in FIG. 5. This is an important feature of the
apparatus 10 of the present invention. Since the large diameter column tubes
processed by the apparatus of the present invention are heavy even when empty
and difficult to maneuver and position over lower **pistons** and bed supports.
Furthermore, insertion of tightly fitting bed supports requires significant
force. With the apparatus of the present invention however, all such lifting
and positioning is accomplished by the hydraulic system of the compression
mechanism 22.

Detailed Description Text - DETX:

Once the column tube 35 is seated on the lower **piston** and bed support 100 as depicted in FIG. 5, the connecting pin 98 is removed from the column tube 35 and coupler 78 and the compression rod and **piston** are allowed to move upwardly out of the upward end of the column tube to the position shown in FIG. 6. The upper open end of the column tube 35 is then clear to allow a pouring of a slurry of packing material into the column tube for compression by the compression **piston** 26 after an upper bed support is inserted into the column tube.

Detailed Description Text - DETX:

Thus, as described above the present invention overcomes the short comings of prior column packing apparatus by providing an apparatus which is vertically collapsible for packaging, shipment and relocation from one location to another in a laboratory. Also, the apparatus of the present invention provides for the lifting and positioning of empty column tubes and the positioning of lower **pistons** and bed supports therein without requiring operators to manually lift, locate and lower the column tubes. Further, the apparatus of the present invention is adapted to compact columns of different diameter by use of adaptor rings and compression **pistons** of different size which are readily mounted and removed from the apparatus which in packing operation repeatedly uses the same compression mechanism in the packing of such columns.

Detailed Description Text - DETX:

While a particular embodiment of the apparatus of the present has been described herein, changes and modifications in the

illustrated embodiment may
be made without departing from the spirit or scope of the
present invention.
For example, the apparatus may be inverted such that the
compression mechanism
is mounted below the column tube with the compression
piston entering a lower
open end of the tube to compress packing material as it is
driven upward into
the tube. Alternatively, the apparatus may be positioned
horizontally rather
than vertically. Accordingly, it is intended that the
scope of the present
invention be limited only by the terms of the following
claims.

Claims Text - CLTX:

a compression piston at an end of the compression rod, and

Claims Text - CLTX:

a drive for moving the compression rod to longitudinally
reciprocate the
compression piston, the compression piston being sized to
fit snugly into one
end of a column tube supported on the first support member
to compress a
packing material within a second end of the column tube
when the movable
support member is secured to the second support member and
the drive is
actuated to drive the compression rod toward the column
tube.

Claims Text - CLTX:

means for releaseably connecting the compression piston to
the column tube to
raise and lower the column tube when the apparatus is
oriented in a vertical
direction with the first support member below the second
support member and
when it is desired to insert a lower end piston into or
remove a lower end

piston from the second end of the column tube or when it is desired to change
an adaptor ring on the first support member to receive a column tube of a
different diameter for packing by the apparatus.

Claims Text - CLTX:

the second support member concludes an opening for receiving the **drive** of the
compression mechanism; and

Claims Text - CLTX:

7. The apparatus of claim 6 wherein the compression **piston** includes an annular
fluid seal for tightly engaging an inner surface of the column tube and the
column tube includes a second **piston** for bearing against an enclosure for the
column tube, the second **piston** including an annular fluid seal for tightly
engaging an inner surface of the column tube.

Claims Text - CLTX:

a compression **piston** at a lower end of the compression rod, and

Claims Text - CLTX:

a **drive** for vertically moving the compression rod to raise and lower the
compression **piston**, the compression **piston** being sized to fit snugly into an
open upper end of the column tube to compress a packing material within a lower
end of the column tube when the **drive** is actuated to **drive** the compression rod
in a downward direction into the column tube; and

Claims Text - CLTX:

means for releaseably connecting the compression **piston** to the column tube to
raise and lower the column tube when it is desired to insert a lower end **piston**
into or remove a lower end **piston** from the lower end of the column tube or when
it is desired to change an adaptor ring on the lower support to receive a
column tube of a different diameter for packing by the apparatus.

Current US Original Classification - CCOR:

210/198.2

Current US Cross Reference Classification - CCXR:

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DATE FILED: September 25, 1990

---------- KWIC ---------

Abstract Text - ABTX:

A liquid chromatography column that can be rapidly packed without a reservoir column is disclosed. The column includes a double sealed compressor **piston** with a mixture delivery bore extending through the body of the **piston**. Resilient sealing means are provided in annular grooves provided in the **piston**. The **piston** retains a frit and slurry scraper is mounted to the **piston**. A method of packing such a column is also disclosed. An outlet end plate including solvent drainage bores is provided. A threaded end cap including radial slots in an inner surface for a threaded column is disclosed, as is an adaptor for joining flanged and threaded columns.

Brief Summary Text - BSTX:

The present invention relates to the field of liquid chromatography. In particular, the present invention is directed to an improved apparatus for use

in obtaining packed and pressurized slurry beds employed in liquid chromatography columns, as well as novel and improved **piston** compressors and end closure members for use in such liquid chromatography columns. The present invention is also directed to a method for packing liquid chromatography columns.

Brief Summary Text - BSTX:

A critical feature in ensuring the proper operation of such columns relates to the degree and uniformity of compactness of the packed absorbent bed contained within the column. The slurry bed must be maintained at very high pressure, and at uniform density, in order to achieve the most efficient end results during the chromatographic procedure. The present invention relates to the configuration and structure of columns for use in preparative liquid chromatography. The present invention also relates to an apparatus for ensuring the rapid and uniform packing of slurry beds. The present invention enables such columns to be uniformly packed, while at the same minimizing the time necessary for packing the column. The present invention also relates to a novel **piston** compressor for use within the chromatography column that is particularly designed to prevent the leakage of particles past the compressor, while at the same time permitting the injection of a material to be separated through the **piston** into the compressed bed. The **piston** compressor of the present invention is utilized to apply a packing compression force to the slurry bed within the column during packing of the column, and, during use of the column in the separation process, is retained within the column. By use of the **piston** compressor of the present invention, the need

for a separate
reservoir column is eliminated.

Brief Summary Text - BSTX:

The present invention also relates to an end plate for use with a
chromatographic column. In particular, the present invention relates to an
outlet end plate which is provided with drainage apertures to enable the
solvent to drain from the slurry while the slurry is being compressed by the
application of fluid pressure to the compressor **piston**. Drainage plugs are
provided for sealing these drainage apertures or openings during the
utilization of the packed column in the separation process.

Brief Summary Text - BSTX:

The present invention further relates to a method of packing and using a column
including the novel **piston** compressor and end plate of the present invention.
The method of the present invention enables columns to be packed efficiently
and in a very short time period. It also enables a large number of columns to
be prepared with a single packing apparatus, and avoids the need for a separate
reservoir column to pack the chromatography column.

Brief Summary Text - BSTX:

The chromatography column of the present invention includes a compressing
piston, movable, by the application of solvent pressure exerted thereagainst,
within the bed, which is maintained under dynamic compression via the **piston,**
during column operation. Thus, the present invention allows the user to pack
any desired stationary phase quickly, and then enables the packing instrument

(fluid/solvent pump) to be utilized to pack additional columns quickly and efficiently. According to the present invention, the desired packing material is transferred into the column, via the outlet end, with the **piston** in its lowest position (i.e., adjacent the inlet end). After the column outlet end plate is attached, the **piston** is raised (i.e., pushed into the packing material) by hydraulic pressure applied by the packing instrument, to pack (compress) the material. The applied hydraulic pressure is retained between the back face of the **piston** and the inlet plate of the chromatographic column. Thus, after packing, the compressor **piston** of the present invention maintains a dynamic pressure on the packed particle bed which is greater than the pressure of the mobile phase flowing through the column. The dynamic action of the **piston** prevents the formation of any undesirable voids or channeling due to bed shrinkage.

Brief Summary Text - BSTX:

Thus, columns packed according to the present invention provide total control of the packing process. Columns packed according to the present invention are very efficient. High pressures can be utilized in columns packed according to the present invention and very small sized particles can be employed at high flow rates. Thus, very high material throughputs are achievable. The constant **piston** pressure that is maintained upon the packed bed during use of the column minimizes and/or eliminates the formation of voids and channeling within the column. Further, excellent reduced plate height values are obtained using the column of the present invention. The packing and unpacking of the column is very simple and quite rapid. The present invention thus

simplifies the
procedure for packing columns, and, because of its
simplicity, speed and
moderate cost, makes high quality liquid chromatography
accessible to a greater
class of users.

Brief Summary Text - BSTX:

The chromatography column of the present invention
comprises an inlet plate, a
double sealed compressor **piston** sealingly received within
the column body,
coiled tubing connecting the inlet end plate with the
double sealed compressor
piston and an outlet end plate. Appropriate porous frits
and sealing O-rings
are also provided as part of the column assembly. The
inlet end plate is
provided with three threaded openings, one of which is
equipped with a valve
and connector for connection to a pushing solvent delivery
system. The valve
is closed, upon disconnection of the pushing solvent
delivery system, to
maintain the pressure within the chromatography column.
The central opening in
the inlet end plate is connected on one side to the double
sealed **piston** via
the tubing, and on the other side to a supply feed line to
enable the injection
of samples or mixtures into the chromatography column
through the inlet end
plate, through the coil tubing, the double sealed **piston,**
and finally into the
packed bed. The third opening in the inlet end plate is
equipped with a liquid
gauge and valve to enable monitoring, maintaining, and
releasing of the
pressure in the chromatography column. The inlet end plate
further includes a
plurality of apertures or bores for receiving therein bolts
which cooperate
with appropriate nuts and washers to assist in mounting and
sealing the inlet
plate, via an appropriate O-ring, to an apertured flange
provided on the column

body.

Brief Summary Text - BSTX:

The coiled tubing is equipped with tubing connectors at each end. The tubing connectors enable the tubing to be sealingly connected to the **piston** and to the inlet end plate. As, during packing of the column, the **piston** is driven by the solvent delivery system down into the column, the tubing must be long enough to extend the length of the column.

Brief Summary Text - BSTX:

The column is a standard chromatography column formed, for example, of stainless steel and having a smooth and accurately ground interior diameter. The column should be large enough to contain enough slurry in its looser, unpacked state so as to yield a packed bed of the required size therein, as well as to contain the compressor **piston** therein.

Brief Summary Text - BSTX:

The compressor **piston** is a significant feature of the present invention. The **piston** includes a generally cylindrical **piston** body and two annular grooves, formed in the outer, peripheral, cylindrical surface of the **piston** body and spaced from each other along the length of the **piston** body and within which O-rings and backup rings are positioned to seal the **piston** against the interior surface of the column body. The front end face (towards the packed bed) of the **piston** is provided with a plurality of radially extending slots or grooves to enable and enhance a uniform dispersal of the material to be separated across

the entire cross-sectional width of the column. A frit is retained within a frit holder which is attached to the front end face of the **piston** and a slurry scraper is secured to the outer perimeter of the frit holder.

Brief Summary Text - BSTX:

Operating the chromatography column of the present invention to pack a compressed bed is very easily performed. First, the **piston** and its components, including the various sealing members, the frit holder and the slurry scraper, are assembled, and the **piston** is inserted into the column, by use of an adapter member, in a position closely adjacent to the inlet end plate. The central opening of the inlet end plate is then connected via the tubing to the **piston**. Then, after an appropriate O-ring is installed in the column flange, the inlet end plate is secured to the column body via nuts and bolts extending through the mating apertures in the flange of the column and in the apertures of the inlet end plate. The slurry is loaded into the column on top of the **piston** until the column is almost full (an air bubble must be left to enable proper mixing). The outlet end plate is then attached by appropriate nuts and bolts and the drainage plugs are removed therefrom. The airpump driven high pressure solvent delivery system attached to one of the threaded openings in the inlet end plate is then actuated to rapidly **drive the piston** down into the slurry loaded into the column, to compress the slurry and to expel the solvent from the open drain bores. After all the solvent has drained out of the drain bores and the column is allowed to equilibrate for an appropriate period of time, the valve connecting the inlet end plate to the liquid delivery system is closed so

that the pressure is retained within the column between the rear face (i.e., the surface facing the inlet end plate) of the **piston** and the inlet end plate. The drain plugs are then inserted into the drain bores in the outlet end plate, and the packed column can then be disengaged from the high pressure solvent delivery system. After connection of the packed column to a chromatography pump to deliver the mobile phase material, chromatography can be performed.

Brief Summary Text - BSTX:

The present invention relates to a slurry packing compressor for use in a chromatographic column which is adapted to contain a bed of particles comprising a slurry. The compressor includes a generally cylindrical **piston** including structure for receiving a resilient means adapted to seal an interface of a chromatography column and the **piston**. A material to be separated is adapted to be injected into the bed of particles, and a mechanism is provided for dispersing the material to be separated throughout the cross section of the column. The compressor also includes a device for retaining a frit.

Brief Summary Text - BSTX:

The **piston** of the invention further includes a packed-bed facing end-surface, a pressure-receiving end-surface, and a cylindrical body portion extending between the end-surfaces. The structure for receiving the resilient means comprises a plurality of annular grooves provided in the cylindrical surface of the **piston**, each groove adapted to receive a resilient sealing means. Further, each resilient sealing means comprises a resilient O-ring

and backup spacer
member. Further, each backup spacer member comprises a split ring glass loaded
polymer. Each resilient O-ring is positioned within each groove to be closer
to the packed bed facing end-surface of the **piston** than the backup spacer
member.

Brief Summary Text - BSTX:

According to another feature of the invention, the **piston** includes a threaded
portion adjacent to the packed-bed facing endsurface of the **piston**, and frit
retaining device is adapted to threadedly engage the threaded portion. The
frit retaining device is capable of receiving a frit sealing member. The frit
retaining device further comprises structure for mating with a tool for
tightening and loosening the frit retaining device with respect to the **piston**.

Brief Summary Text - BSTX:

According to another feature of the invention, the material to be separated is
injected into the slurry bed via a bore extending through the **piston**. The bore
sealingly mates with a supply line for delivering a material to be separated
through the **piston** into a packed bed retained in the column..

Brief Summary Text - BSTX:

According to another feature of the invention, a mechanism is provided for
ensuring the uniform dispersal of a material to be separated throughout the
column.. The mechanism comprises a plurality of radially extending slots or
grooves disposed in the packed bed facing end-surface of the **piston**.

Brief Summary Text - BSTX:

The present invention also relates to a chromatography column assembly including a column comprising inlet and outlet ends, the interior of the column comprising a chamber adapted to retain a packed slurry bed. The column assembly further includes an inlet end plate and an outlet end plate, the end plates including a mechanism for enabling the plates to be sealingly secured to the column. Further, the assembly includes a compressor **piston** adapted to be positioned within the chromatography column and including structure for ensuring leakproof engagement between the **piston** and the interior of the column. Structure is connected to the inlet end plate and extends through the compressor **piston** for delivering a material to be separated into the packed slurry bed. Further, a mechanism is provided for draining the slurry solvent from the chamber through the outlet end plate. The assembly also contains means for retaining a frit adjacent the end of the **piston** facing towards the packed slurry bed.

Brief Summary Text - BSTX:

The inlet end plate further includes a mechanism for enabling the application of hydraulic pressure to the **piston** to pack the slurry bed and for maintaining **piston** pressure during use of the column for separation. The pressure application mechanism comprises disconnectable means for exerting a hydraulic pressure against the compressor **piston** and a mechanism for retaining the exerted pressure after disconnection of the exerting means.

Brief Summary Text - BSTX:

Further, the outlet end plate comprises a plurality of solvent drainage apertures or bores which comprise the draining mechanism. The compressor **piston** further includes structure ensuring the uniform dispersal of a material to be separated throughout said column. The compressor **piston** includes a plurality of radially extending slots or grooves disposed in an end surface of the **piston** facing towards the packed slurry bed, with the slots comprising the structure for ensuring the uniform dispersal of a material to be separated throughout the column. The structure for ensuring leakproof engagement between the **piston** and the column includes a plurality of annular grooves provided in a cylindrical surface of the **piston**, each groove adapted to receive a resilient sealing mechanism. Further, each resilient sealing mechanism includes a resilient O-ring and a backup spacer member.

Brief Summary Text - BSTX:

The present invention also relates to a method of packing a liquid chromatography column having means for securing inlet and outlet end plates to the flanged ends of the column. The method comprises positioning a compressor **piston** having first and second end faces within the column, connecting a material supply line between an inlet end plate and a second end face of the **piston**, securing the inlet end plate to the column, and substantially filling the column above the first end face of the **piston** with a slurry. The method further includes securing an outlet end plate to the column, applying pressure, from a pressure source, to compress the slurry between the first end face of

the compressor **piston** and the outlet end plate, allowing the slurry solvent to drain through the outlet end plate, and sealing the end plate after the solvent has been drained from the compressed slurry.

Brief Summary Text - BSTX:

The method further comprises the step of retaining a frit adjacent a first end face of the **piston**, and supplying a material to be separated within the compressed slurry through the **piston**. The pressure between the inlet end plate and the **piston** second face is retained after disconnection of the column from the source of pressure, and a plurality of annular, resilient, seal-containing formations are provided between the first and second faces of the **piston**.

Brief Summary Text - BSTX:

The method of the present invention further includes uniformly dispersing a material to be separated throughout the column. Ensuring the uniform dispersal of a material to be separated involves disposing a plurality of radially extending slots (or grooves) within a first end surface of the compressor **piston**. The present method further includes the provision of a slurry scraping member attached adjacent the first end face of the compressor **piston**.

Drawing Description Text - DRTX:

FIG. 2A-2E are various views of parts of the compressor **piston** of the present invention, Wherein:

Drawing Description Text - DRTX:

FIG. 2(a) is an end view of a first inner packed-bed contacting end face of the **piston**;

Drawing Description Text - DRTX:

FIG. 2(b) is a sectional side view of the scraper of the compressor **piston** of the present invention;

Drawing Description Text - DRTX:

FIG. 3(d) is a sectional side view of the body of the compressor **piston** of the present invention;

Drawing Description Text - DRTX:

FIG. 4 is a side view, on a greatly enlarged scale, of a center aperture of the general type provided in the inlet and outlet end plates, as well as in the **piston**, showing the shape and threading thereof adapted to threadably receive a tubing connector;

Detailed Description Text - DETX:

With reference to the drawings and in particular with reference to FIG. 1, there is illustrated, in sectional side view, a self slurry packing column according to a preferred embodiment of the present invention. The column includes a column body 100, having an inlet flange 120 and an outlet flange 110 rigidly secured thereto. Each of the inlet and outlet flanges is provided with a plurality of apertures 130 disposed about the periphery thereof, by means of which corresponding inlet and outlet end plates 300 and 400 can be secured to the column body 100. The column body 100 is formed of stainless steel or other

suitable chemically resistant material and the inner diameter of the column body is smoothly finished so as to be able to sealingly receive therein the compressor **piston** 200 of the present invention. The column body 100 can be formed with any desired diameter and should be of a length large enough to contain the **piston** as well as to contain enough slurry material to form a packed bed of the desired length.

Detailed Description Text - DETX:

central opening 340 of the inlet end plate 300, as most clearly illustrated in FIG. 3(c) and is equipped with two tubing connectors 380, 370 mounted to extend in opposite directions. A teflon plug 350 is disposed between the tubing connectors and is provided with a small diameter hole 360 bored through the center of the plug 350 to enable a mixture to be separated to pass through the inlet end plate of the system, and enables the delivery of such mixtures through the inlet end plate into the packed bed contained within the chromatography column for analysis and separation. Thus, the outer tubing connector 380 is connected to an appropriate supply of a chemical mixture to be separated, while the inner tubing connector 370 is connected to the compressor **piston** which forms a further feature of the present invention.

Detailed Description Text - DETX:

Connected to the inwardly extending end of the tubing connector 370 is a length of coiled tubing 331 which extends from the tubing connector 370 to a generally similar tubing connector 332 threadably engaged to the **piston** 200. The coiled tubing, which can be made of stainless steel or any other

similar chemically
resistant material, connects the **piston** at one end and the inlet end plate at
the other, and enables a material mixture or sample to be fed to and through
the compressor **piston** body. For reasons that will become clear later, the
length of the tubing must be long enough to extend substantially over the
entire length of the column body.

Detailed Description Text - DETX:

The **piston** compressor 200, which forms a significant feature of the present
invention, is a double-sealed, slurry packing **piston** made of assembled
components shown in FIG. 1 in their assembled conditions, and in greater detail
in FIGS. 2(a), 2(b), 2(c), 2(d) and 2(e). The compressor **piston** 200 of the
present invention can typically be formed of stainless steel or a similar
chemically resistant material, and comprises a **piston** body 310 as shown most
clearly in FIG. 2(d). The **piston** body is of generally cylindrical shape,
having a first end face 336, which, when installed in the column, faces the
inlet end plate 300. The **piston** body 310 also includes a second end face 338
that faces the compressed bed of slurry material. Intermediate the end faces
336 and 338, spaced first and second annular grooves 340 and 350 are provided
in the cylindrical surface of the **piston** body 310. Adjacent the end face 336 a
necked down portion containing threads 354 is provided. At the center of the
end face 336, a threaded aperture for a tubing connector is provided. Into
this threaded aperture, a tubing connector 332, which is connected to the
coiled tubing 500, is engaged. The threaded opening 330 continues through the
piston body as a relatively small bore 320, through which

the mixture to be
separated passes to reach the frit retained adjacent the
end face 338.

Detailed Description Text - DETX:

Within each of the two wide annular grooves 340, 350, an
O-ring 342 and a
backup spacer member in the form of a flat, split ring 344
are positioned. The
O-ring 342 is formed of a Fluorez (or any other chemically
resistant) material,
while the split ring 344 is typically formed of glass
loaded TFE or a similar
polymeric material. Within each groove 340, 350, the
O-ring 342 is mounted
closer to the slurry contacting end face 333 of the **piston**
compressor 200,
while the split ring 344 is mounted closer to the pressure
receiving end face
336.

Detailed Description Text - DETX:

The frit holder 360, shown in a cross sectional side view
in FIG. 2(c), is
mounted to the **piston** body 310. The frit holder 360 is
provided with a first
interior threaded portion 362 which is designed to matingly
engage with the
threads 354 on the necked down portion of the **piston** body
310. A second
externally threaded portion 364 is provided about the outer
surface of the frit
holder. As shown most clearly in the enlarged view of FIG.
2(e), the frit
holder 360 is shaped such that an O-ring groove 368 is
provided therein. A
conventional TFE O-ring can be inserted into the groove 368
to seal the frit
holder 360 against the **piston** body 310. The frit holder
can be manufactured
from stainless steel and should be silver plated to avoid
galling or the
premature wearing away of the threads 362 thereon by
friction between the

threads 362 and the mating threads 354, provided on the **piston** body 310 and
which can both typically be formed of stainless steel.

Detailed Description Text - DETX:

On an outwardly extending end face of the frit holder 360, a plurality,
typically four or more, recesses 366 are formed. An appropriately designed and
constructed spanner wrench or other pronged tool is provided, having pins which
are positioned to mate with the recesses 366 to enable sufficient torque to be
applied to the frit holder 360 to enable the frit holder to be tightly mounted
on and demounted from the face 338 of the **piston** 310. By properly tightening
down the frit holder against the **piston** surface, the frit will be tightly
retained thereagainst, and, because of the O-ring positioned within the space
364, no packing media will be able to penetrate into the feed line 320.

Detailed Description Text - DETX:

The frit (non-illustrated) for the **piston** is designed and sized to fit (i.e.,
thickness and perimeter) between the frit holder 360 and the face 338 of the
piston. The frit holder is manufactured of a porous stainless steel or other
porous material as is conventional in the chromatography technology. The
porosity of the frit should match (i.e., be smaller than) the particle size of
the packing media.

Detailed Description Text - DETX:

The scraper 370, formed of TFE or a similar polymer, is provided to prevent the
typically stainless steel **piston** body 310 from possibly scraping and thus

damaging the machined stainless steel interior surface of the column body 110.
The scraper also serves to move the packing media ahead of the **piston,** and to
keep it there so that it does minimum damage to the sealing O-rings on the
piston. Further, the scraper prevents grinding and crushing of the packing
media between the stainless steel **piston** and the stainless steel column body.

Detailed Description Text - DETX:

As shown in FIG. 2(a), the end face 338 of the **piston** body 310 is provided with
a plurality of radially extending slots or grooves. One groove 390 is shown in
FIG. 2(a); however, typically 24 or more such grooves are provided in an
arrangement similar to that shown in FIG. 10(a) and extending from the central
aperture 320 of the **piston** radially outwardly therefrom to a position adjacent
to the peripheral end of the face 338. These radial slots or grooves 390 serve
to uniformly disperse the incoming mixture to be separated throughout the cross
sectional area of the column. The mixture to be separated is injected via an
appropriate supply mechanism, through the supply line 399 extending through the
central aperture 340 of the inlet end plate 300, through the tubing 500,
through the bore 320 in the compressor **piston,** through the radial grooves 390,
and finally through the frit into the packed slurry bed. The grooves, radially
disposed within the end face 338, increase in width and depth as they radiate
outwardly from the center of the end face 338, forming a dispersion chamber
integrally structured within the end face of the **piston** to facilitate prompt
and uniform distribution of incoming chemicals.

Detailed Description Text - DETX:

FIG. 4 shows, in a greatly enlarged detail, the shape and structure of the threaded opening 330 provided in the end plate 336 of the compressor **piston** body 310. Generally similar threaded openings are provided in both the inlet end plate 300 and the outlet end plate 400. As shown in FIG. 4, the threaded opening 330 is provided with a plurality of threads 352 and a tapered section 356 which culminates in a smaller diameter aperture which leads to the narrow bore 320 extending the length of the **piston**. The threads 352 are designed to accept an appropriate mating quick connect coupling for enabling the connection of tubing, such as coiled tubing 500 to the **piston**.

Detailed Description Text - DETX:

As can be understood, during the packing of the chromatography column of the present invention, the packing drainage plugs 450 are removed from the bores or apertures 430 so as to enable the slurry packing solvent to drain freely from these apertures as pressure is applied to the slurry material by the **piston** 310. After the column is tightly packed, these plugs are installed therein and tightened until the surfaces 458 are flush with the interior surface of the outlet end plate 400, so that the separated compounds will flow out through the center opening 460 only.

Detailed Description Text - DETX:

The flanged end plate chromatography column illustrated in FIGS. 1 through 7 can be packed in a novel manner according to a method of the present invention. First, the **piston** is assembled. In other words, the flat and circular O-rings 342 and 344 are assembled into each of the annular grooves

340 and 350. A
further O-ring is positioned within the O-ring groove 368 that is provided
within the frit holder 360. A frit is installed on the face 336 of the **piston**
body, and the frit holder 360 is threadedly mounted thereon. An appropriate
tool having pins that mate with the holes 366 is utilized to tightly secure the
frit holder and the frit carried thereby onto the **piston** body. The slurry
scraper is then threadedly engaged over the frit holder.

Detailed Description Text - DETX:

Connector 332 with coiled stainless steel tubing 500 is then inserted into the
central threaded aperture 330 of the **piston** body. The other end of the tubing
510, having a similar threaded connector 370, is inserted into the inwardly
facing central aperture of the inlet end plate 300. The **piston** is then
inserted into the body of the column as shown in FIG. 1 by means, for example,
of the adaptor member disclosed in co-pending, commonly assigned U.S. Ser.
No. 07/501,122 filed on Mar. 29, 1990 in the name of Dr. Y. Shalon, the entire
disclosure of which is expressly incorporated herein by reference.

Detailed Description Text - DETX:

Thereafter, using an O-ring and the appropriate nuts, bolts and washers, the
inlet end plate 116 is securely attached to the column flange 120. The region
110 between the rear surface of the **piston** 336 and the front (i.e., inward)
surface of the inlet end plate can then be filled with a pushing solvent at
atmospheric pressure, and the apertures 330 and 320 closed by means of the
associated valves provided in the couplings threadedly attached to these

apertures.

Detailed Description Text - DETX:

The column should then be mounted in an appropriate stand with the open end (where the outlet end plate will eventually be secured) facing upwardly. Thus, the inlet end plate and **piston** will be at the bottom of the column. The column interior 112 should then be filled with a slurry, up to within several centimeters of the top. By leaving a space at the top of the column, an air bubble will be formed which will aid in the mixing and the maintenance of a uniform dispersion of packing material within the column. The outlet end plate is then assembled onto the top of the column. The assembling of the outlet end plate onto the upper end of the column involves placing the bed support frit in the frit recess of the column body. The disperser frit and small TFE O-ring are then placed on top of the bed support frit, and the large TFE O-ring is positioned within the flange groove. The outlet end plate is the secured to the outlet flange 110 of the column body 100, with the aid of the nuts, bolts and washers as is conventional and as is disclosed with great particularity in the aforementioned U.S. Pat. No. 4,882,047. After the outlet end plate is securely fastened to the flange 110, the column can be rotated (i.e., inverted) several times to aid in mixing of the slurry. Complete mixing of the slurry should take place within several minutes.

Detailed Description Text - DETX:

The column should now be inverted within the holder so that the inlet portion of the column is up, and an appropriate solvent pushing instrument should be

attached to the aperture 320 via the appropriate quick connector. The solvent delivery system generally uses an air-driven, high pressure pump which is capable of delivering the solvent through the hose fitting and valve into the space 116 provided between the inlet end plate 300 and the end face 336 of the **piston**. The packing drain plugs 450 have been previously removed from the drainage bores 430 of the outlet end plate, and, as the pump rapidly pushes the **piston** down at the preset pressure, the solvent within the slurry in the area 112 of the column will be ejected through the drainage bores 430. After several minutes, when no more solvent is observed to be draining out of the drainage bores 430, the packing of the column has been completed and the column should be allowed to equilibrate for several more minutes.

Detailed Description Text - DETX:

The valve connecting the solvent delivery pump to the inlet end plate aperture 320 can now be closed to retain the fluid pressure within the column portion 116 acting on the **piston** face 336 to maintain the compressed slurry bed in the volume 112 at the appropriate pressure. The packing drainage plugs 450 should now very quickly be inserted into the bores 430; and after attaching the slurry packing chromatography column of the present invention to the appropriate source of a fluid mixture to be separated, the column can be used.

Claims Text - CLTX:

a generally cylindrical metallic **piston**;

Claims Text - CLTX:

means for receiving resilient means adapted to seal an interface of said chromatographic column and said **piston**;

Claims Text - CLTX:

means for dispersing the material to be separated throughout the chromatography column, said dispersing means comprising a plurality of radially extending grooves provided in a slurry facing end surface of said generally cylindrical metallic **piston**; and

Claims Text - CLTX:

means for retaining a frit comprising a threaded portion, said **piston** including a threaded portion adjacent the slurry contacting end face of said **piston** and adapted to engage said threaded portion of said frit retaining means, said frit retaining means further comprising means for receiving a frit sealing means.

Claims Text - CLTX:

2. The slurry packing compressor according to claim 1, said receiving means comprising a plurality of annular grooves provided in a cylindrical surface of said **piston**, each said groove adapted to receive resilient sealing means.

Claims Text - CLTX:

3. The slurry packing compressor of claim 3, wherein said resilient O-ring is positioned within each said groove to be closer to a slurry bed facing end surface of said **piston**.

Claims Text - CLTX:

6. The slurry packing compressor of claim 3, said **piston** further comprising
means for applying a hydraulic packing pressure against a slurry in said
column..

Claims Text - CLTX:

7. The slurry packing compressor of claim 6, said **piston** further comprising
means for maintaining a packed slurry bed during use of said column for
separation.

Claims Text - CLTX:

8. The slurry packing compressor of claim 1, said frit means further
comprising means for mating with a tool for tightening and loosening said frit
retaining means with respect to said **piston**.

Claims Text - CLTX:

11. The slurry packing compressor of claim 1, said means for enabling
comprising a bore extending through said **piston**.

Claims Text - CLTX:

12. The slurry packing compressor according to claim 11, said bore comprising
means for sealably mating with a supply line for delivering material to be
separated through said **piston** into a packed bed retained in said column.

Current US Original Classification - CCOR:

210/198.2

Current US Cross Reference Classification - CCXR:

210/656

US-PAT-NO: 5167809
DOCUMENT-IDENTIFIER: US 5167809 A

TITLE: Chromatography system

DATE-ISSUED: December 1, 1992

US-CL-CURRENT: 210/198.2; 210/189 ; 210/656 ; 96/101

APPL-NO: 07/ 757868

DATE FILED: September 11, 1991

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
GB	9020453	September 19, 1990

---------- KWIC ---------

Abstract Text - ABTX:

A chromatography system comprises a chromatography column 1 consisting of a
column tube 7 having end flanges 19 and 27 at its lower and upper ends,
supporting an upper end cap 3 carrying a **piston** rod 9 and a **piston** 11. The
system further comprises a container 20 for the separation media used in the
chromatography column, this container having a neck ring 24 of injection
moulded construction with a mouth end of a bag 22 embedded therein.

Detailed Description Text - DETX:

The column further comprises a **piston** rod 9 carrying a **piston** 11 which serves
as upper end cell for the chromatography bed and through which the medium to be
separated can pass, after introduction through the hollow

interior 13 of the
piston rod 9. The preferred construction of the piston of this bed is
described and claimed in our simultaneously filed British Patent Application
No. 9020450.4.

Detailed Description Text - DETX:

The piston rod 9 can be clamped by a friction clamp or adjusted by a screw
action, by virtue of the manner in which the piston rod is mounted in the upper
end cap 5. This preferred mechanism is described and claimed in our
simultaneously filed British Patent Application No. 9020451.2.

Detailed Description Text - DETX:

The upper end cap furthermore preferably includes a pneumatic inlet 15 to allow
pneumatic driven movement of the piston 11, as is described in our said British
Patent Application No. 9020451.2.

Detailed Description Text - DETX:

When the bed is to be unpacked, the clamp (not shown) holding the lower end cap
5 in place is removed allowing the end cap 5 then to be withdrawn from the
bottom of the column. The chromatography media in the bed space 17 can then be
discharged, preferably with the aid of pneumatic pressure applied through the
inlet 15 after release of the friction clamp on the piston rod 9. This allows
the piston 11 to be displaced downwardly to displace the separation media in
turn from the bed space 17 into a container shown in more detail in FIGS. 2 to
4.

Detailed Description Text - DETX:

Upon re-packing of the bed, the lower end plate 5 will have been replaced and clamped in position by its appropriate clamp, and the upper end cap 3 will have been removed by release of its clamp, and by withdrawal of the **piston** 11 and **piston** rod 9 from the interior of the column tube 7. This allows the separation media to be introduced, either by pouring into the bed in the case of fresh separation media of a relatively easily handled form, or by introduction from a container of the type just described for use in receiving the discharged separation media on unpacking; this recharging operation will be described later with reference to FIG. 3.

Detailed Description Text - DETX:

The state of the separation media 23 in the column will be that of a relatively self-supporting mass which has been packed in place by virtue of both the binding action of the liquid which has just passed through the column, and the fact that the column may well have been pressurised during the chromatography operation. It is therefore necessary to lower the **piston** 11 by driving the **piston** rod 9 downwardly to expel the separation media 23 into the container 20. If desired this may be achieved manually, but it is preferred to expel the separation media pneumatically by releasing the friction clamp and applying pneumatic pressure through the inlet 15 to **drive the piston** downwardly thereby expelling the separation media 23 from the column and into the container 20.

Detailed Description Text - DETX:

Upon completion of the discharge stroke of the **piston** 11 to

arrive at the
bottom end of the column tube 7, either that stroke can be terminated or, by
virtue of the mating action of the container neck column 24 on the lower end
flange 19 of the column, the stroke may be continued in order to **drive** the
separation media clear of the neck collar 24, positively into the bag portion
22 of the container 20. If this latter additional stroke has been effected,
the **piston** 11 will need to be retracted before the container 20 can be removed.

Detailed Description Text - DETX:

When re-packing is required using media already contained in a container 20,
the column 1 on which the lower end cap 5 has already been replaced, as shown
in FIG. 4, has its upper end cap 3 and **piston** structure removed and then, after
removal of the snap-on cover 26 from the neck collar 24 of the container 20,
the container is secured in place by clamping of its neck collar 24 to the
upper end flange 27 of the column tube 7, using the upper clamp 29.

Detailed Description Text - DETX:

Once the clamp 29 has been fastened the upper end of the bag can be pushed down
manually, again by grasping the hand-hold aperture 34, to expel the separation
media from the container 20 into the interior of the chromatography column tube
7. Once all or substantially all of the separation media is in the column,
preferably with all of it below the neck collar 24, the clamp 29 can be
unfastened in order to allow the neck collar 24 of the container 22 to be
removed from the upper flange 27, and then to allow replacement of the upper
end cap 3 with its **piston** 11 and **piston** rod 9.

Detailed Description Text - DETX:

Once the container neck collar 24 has been removed, the snap-on cover 26 is replaced on the container to keep its interior sterile, and thereafter, once the upper end cap 3 and its **piston and piston** rod structure have been replaced, the **piston** can be traversed downwardly to **drive** the separation media down against the lower end cap 5, or more properly against the lower end cell carried thereby, and the bed may if necessary be pressurised before start of the next separation operation by introduction of in-feed material into the bed through the **piston** 11.

Claims Text - CLTX:

1. A chromatography system comprising a column tube having end flanges for releasable attachment of end caps of the chromatography column, and at least one container having a neck formation adapted to engage said end flanges for attachment of the container to the ends of the column tube, allowing discharge of separation media from the column into such a container upon unpacking, and from such a container into the column upon re-packing, wherein said container includes a neck collar having a form suitable for clamping to the neck flanges of the column tube using clamps which otherwise clamp said end caps in place and wherein said container further comprises a bag having its mouth defined by said neck collar, and **piston** discharge means for discharging media from said column.

Current US Original Classification - CCOR:

210/198.2

Current US Cross Reference Classification - CCXR:

210/656

US-PAT-NO: 5021162
DOCUMENT-IDENTIFIER: US 5021162 A

TITLE: Method for forming a gel bed in a column for liquid chromatography and
an axially adjustable-type column device used for this method

DATE-ISSUED: June 4, 1991

US-CL-CURRENT: 210/635; 210/198.2 ; 210/656

APPL-NO: 07/ 472230

DATE FILED: January 30, 1990

PARENT-CASE:

This is a division of application Ser. No. 07/315,857, filed on Feb. 27,
1989, now U.S. Pat. No. 4,927,531.

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
JP	63-43003	February 25, 1988

---------- KWIC ---------

Brief Summary Text - BSTX:

An example utilizing such a pressurized packing method as disclosed in Japanese
Patent Publication No. Sho 56-20284 is shown in FIG. 6. In FIG. 6, a
piston-like sliding body 3 having a head 9 is fit liquid-tightly and vertically
movably in the column body 10. The liquid collecting plate 13 is attached to
the upper end of the above mentioned head 9. While the sliding body is at the
lower dead point of its travelling path, homogeneous gel slurry 11 is poured

into the column body 10. After that, the liquid dispersing plate 16 and a cover 12 are fixed to a fixing flange 14 on top of the column body.

Brief Summary Text - BSTX:

When the sliding body 8 is moved upward from its lower dead point by a **drive** shaft 15, for example by utilizing a pneumatic or hydraulic jack, pressure is applied to the homogeneous gel slurry 11 to form the gel bed. Removal of the gel bed for replacement is performed by lifting the sliding body 8 with the cover 12 removed. Then it is ready for the next chromatographic operation.

Current US Original Classification - CCOR:

210/635

Current US Cross Reference Classification - CCXR:

210/198.2

Current US Cross Reference Classification - CCXR:

210/656

US-PAT-NO: 4861473
DOCUMENT-IDENTIFIER: US 4861473 A

TITLE: Apparatus for facilitating compression of packing material in a liquid chromatography column

DATE-ISSUED: August 29, 1989

US-CL-CURRENT: 210/198.2
DISCLAIMER DATE: 20021105

APPL-NO: 07/243177

DATE FILED: September 8, 1988

PARENT-CASE:

CROSS-REFERENCE TO RELATED APPLICATION This is a continuation of application Ser. No. 07/055,819 filed on May 29, 1987, now abandoned, which is continuation of application Ser. No. 06/742,265 filed in June 7, 1985, now U.S. Pat. No. 4,670,141, which is a continuation-in-part of a patent application of Carl L. Shackelford and Kenneth Rainin, entitled "Modular Liquid Chromatography Column Apparatus," Ser. No. 584,611, filed on Feb. 29, 1984, now U.S. Pat. No. 4,551,249, and assigned to the same assignee as this application.

---------- KWIC ---------

Brief Summary Text - BSTX:

On the one hand, known liquid chromatography systems, such as the liquid chromatography systems disclosed in U.S. Pat. Nos. 4,283,280 and 4,313,808,

do not apply pressure to the packing material while maintaining seal integrity.
On the other hand, the liquid chromatography column apparatus disclosed in the copending patent application of Carl L. Shackelford and Kenneth Rainin, entitled "Modular Liquid Chromatography Column Apparatus," Ser. No. 584,611, filed on Feb. 29, 1984, and assigned to the same assignee as this application, discloses a modular liquid chromatography column apparatus which applies pressure to the packing material while maintaining seal integrity. Pressure is applied to the packing material by tightening an axial compression nut threadably connected with a threaded column tube such that an axial compression **piston** axially disposed within the axial compression nut is forced against a movable bed retainer in contact with sorbent packing material contained within the tube for compressing the packing material.

Brief Summary Text - BSTX:

In accordance with this invention, a novel and useful liquid chromatography column apparatus is provided which solves the problem encountered in compressing the packing material in liquid chromatography column apparatus. The invention is particularly advantageous when incorporated in modular liquid chromatography column apparatus, especially modular liquid chromatography column apparatus having a relatively large diameter column. In accordance with one embodiment of the invention, compressing means in the form of a manually actuable gear mechanism is incorporated in the end fitting of the liquid chromatography column apparatus for facilitating application of axial force to plugging or enclosing means in order to **drive** the plugging or enclosing means against the packing material, thereby compressing the

packing material so as to
eliminate voids and channels in the packing material.

Detailed Description Text - DETX:

Compressing means 270 additionally includes a **drive** gear 284 having teeth which
mesh with the teeth of pinion gear 276 at a location indicated by the numeral
286 in FIG. 5. Compressing means 270 further includes a threaded spindle 288
threadably engaged in a threaded central bore 290 of housing 274. **Drive** gear
284 is fixedly mounted to spindle 288. Preferably, spindle 288 is provided
with threaded wells 292. **Drive** gear 284 is provided with bores 294. Threaded
wells 292 and bores 294 are aligned, and screws 296 are inserted through bores
294 and rotated into threaded wells 292 for mounting **drive** gear 284 to spindle
288.

Detailed Description Text - DETX:

After pinion gear 276, **drive** gear 284, and spindle 288 are installed in housing
274, end cap 272 is secured to housing 274. Preferably, housing 274 includes
at least one threaded recess 298, and end cap 272 includes at least one bore
300 having a countersink 302. Threaded recess 298 is aligned with bore 300,
and a screw 304 is inserted through bore 300 and rotated into threaded recess
298 for securing end cap 272 to housing 274.

Detailed Description Text - DETX:

Central bores 314, 316, and 318 are provided in end cap 272, **drive** gear 284,
and spindle 288, respectively, so that tubing 319 can be connected in fluid
communication with seal retainer 275, such as by providing tubing 319 with a

threaded ferrule 322 threadably secured in seal retainer 275.

Detailed Description Text - DETX:

In operation, an allen wrench (not shown) is used for rotating allen bolt 310 so that pinion gear 276 rotates in a direction for rotating **drive** gear 284 to initially retract spindle 288 toward end cap 272. Next, housing 274 is grasped, and an internally threaded rim 312 is engaged with the threaded portion of bushing 180, for example. Housing 274 is then rotated by hand for tightening housing 274 onto bushing 180, for example.

Detailed Description Text - DETX:

Thereafter, the allen wrench (not shown) is inserted in allen bolt 310 and operated for rotating pinion gear 276 in a direction which rotates **drive** gear 284 in a direction for extending spindle 288 away from end cap 272. Spindle 288 engages enclosing means 172 having seal retainer 275 incorporated therein, for example, and applies axial force to enclosing means 172 such that enclosing means 172 is driven in a direction opposite the arrow 162 shown in FIG. 2. The axial force is transmitted through enclosing means 172 to packing material 202, as well as through enclosing means 170, coupling element 164, and enclosing means 168 to packing material 204, as well as through bushings 180, 182, and 178, end fitting 198, and enclosing means 166, for example, for compressing the packing material in tubes 150 and 156.

Detailed Description Text - DETX:

Pinion gear 276 has a diameter smaller than the diameter of **drive** gear 284 for

providing substantial mechanical advantage by the use of a small allen wrench.
Consequently, the use of large wrenches, such as used for tightening seals in
known liquid chromatography equipment, is avoided. The substantial mechanical
advantage enables effective elimination of voids and channels in packing
material 202 and 204, for example.

Current US Original Classification - CCOR:

210/198.2

US-PAT-NO: 4470910
DOCUMENT-IDENTIFIER: US 4470910 A

TITLE: Chromatographic apparatus and process for its use

DATE-ISSUED: September 11, 1984

US-CL-CURRENT: **210/656**; **210/198.2** ; 422/70

APPL-NO: 06/ 470027

DATE FILED: February 25, 1983

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
FR	82 03177	February 25, 1982

---------- KWIC ---------

Abstract Text - ABTX:

A liquid-phase preparative chromatographic apparatus comprising a column in the form of a cylindrical tube equipped at its lower section with a cap; a filling device to be connected by means of a compression nozzle and comprising a cylinder for actuating a non-porous solid body that compresses the stationary phase with which the column is filled by means of the compression nozzle; an injection head to be fastened onto the column after it is filled and comprising a lower wall and an injection unit having a diameter smaller than the injection head and equipped with several lateral solute supply ports, the head comprising on its upper surface a central solvent supply port and means enabling the solvent to flow around the injection unit; a pump connected to the injection head and comprising four **pistons** activated by cams or rods

disposed at
90.degree. angles from one another and having a base of support that is not
integral with the block; a detection device consisting of a double detection
assemblage; and a fraction collector connected to the detection means and
comprising two superimposed compartments.

Brief Summary Text - BSTX:

Because of said means, it is possible to improve the performance of the colum.
considerably by avoiding the disadvantages mentioned above. Among other
things, granulometric gradients and the flow of eluates through the **piston** can
be avoided in this way. The efficiency of the column is also considerably
improved. This is a very important criterion, since fineness of separation
depends on efficiency and on the possibility of separating products according
to their variable retention times in the column.

Brief Summary Text - BSTX:

With regard to the circulation of the solvent, the most important problem has
been the pulsing of the pump. The inventors solved this problem by employing a
four **piston**, 90.degree., cam- or rod-activated pump giving a perfectly linear
and completely stabilized output for a given uniform speed of rotation of the
cam-shaft.

Brief Summary Text - BSTX:

The injection head is, according to the invention, connected to a pump that
comprises four **pistons** activated by cams or rods arranged at 90.degree. to one
another, whose support base is not integral with the block comprising the four

pump heads, said block comprising four pump heads each equipped with a suction
valve and a delivery valve not integral with the motor-cam shaft support.

Drawing Description Text - DRTX:

FIG. 5 is a schematic section and projection of the four-**piston** pump for
liquid-phase chromtography;

Detailed Description Text - DETX:

FIG. 5 represents the four-**piston** chromatographic pump, the output of which is
adjustable during operation. A motor 13 that may be electric or pneumatic uses
belt 14 to **drive** cam shaft 15, which is affixed to bearings 16.

Detailed Description Text - DETX:

Drive pistons 17 are not integral with the cams, thus making it possible to
adjust their stroke with reference to the pump frame. Compression pistons 19
are integral with drive pis**tons 17 and a**re equipped with return springs 18.

Detailed Description Text - DETX:

The solvent is compressed within compression chamber 21 by compression **piston**
19. Solvent enters and leaves compression chamber 21 through double
suction-delivery valve 20. The hatched portion of FIG. 5 representing the pump
piston and head block can be translated with respect to the non-hatched portion
representing the motor and cam shaft block by a distance equal in length to the
depth of the compression chamber 21 by means of vernier 22. A screw 23 for
blocking the slides of the block enables the assembly to

lend rigidity to the
adjustment system.

Claims Text - CLTX:

4. Liquid-phase chromatographic apparatus having a chromatographic column
connected to an injection head which is in turn connected to a pump comprising
four **pistons** actuated by cams or rods disposed at 90.degree. to one another,
and further comprising a block, not integral with the supporting base of said
pump, comprising four pump heads each having a suction valve and a delivery
valve not integral with the support for the motor/cam shaft.

Claims Text - CLTX:

(c) a pump connected to said injection head and comprising four **pistons**
actuated by actuation means disposed at 90.degree. angles to each other and
having a base of support that is not integral with a block comprising four pump
heads, each equipped with a suction valve and a delivery valve not integral
with said actuation means;

Claims Text - CLTX:

(e) connecting said injection head to a pump comprising four **pistons** (19)
activated by cams or rods (14) positioned 90.degree. from each other, one of
four pump heads being connected to said central supply port of said injection
head and the three others to said lateral ports;

Current US Original Classification - CCOR:

210/656

Current US Cross Reference Classification - CCXR:

210/198.2

	Comments	Error Definition	Errors
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2			0
3			0
4			0
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9			0
10			0

	Type	L #	Hits	Search Text	DBs	Time Stamp
1	BPS	L1	4484	replac$5 near5 screen	USPAT	2003/03/04 14:48
2	IS&R	L2	1683	(210/198.2).CCLS.	USPAT	2003/03/04 14:48
3	IS&R	L3	1936	(210/656-659).CCLS.	USPAT	2003/03/04 14:49
4	IS&R	L4	750	(210/635).CCLS.	USPAT	2003/03/04 14:49
5	BPS	L5	2555	2 or 3 or 4	USPAT	2003/03/04 14:49
6	BPS	L6	6	1 and 5	USPAT	2003/03/04 14:52
7	BPS	L7	754697	drive	USPAT	2003/03/04 14:52
8	BPS	L8	404	5 and 7	USPAT	2003/03/04 14:53
9	BPS	L9	214835	piston	USPAT	2003/03/04 14:54
10	BPS	L10	116	8 and 9	USPAT	2003/03/04 14:54

	Comments	Error Definition	Errors
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2			0
3			0
4			0
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6			0
7			0
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L Number	Hits	Search Text	DB	Time stamp
1	4484	replac$5 near5 screen	USPAT	2003/03/04 14:48
2	1683	(210/198.2).CCLS.	USPAT	2003/03/04 14:48
3	1926	(210/656-659).CCLS.	USPAT	2003/03/04 14:49
4	750	(210/635).CCLS.	USPAT	2003/03/04 14:49
5	2555	((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)	USPAT	2003/03/04 14:49
6	6	(replac$5 near5 screen) and ((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.))	USPAT	2003/03/04 14:52
7	754697	drive	USPAT	2003/03/04 14:52
8	404	(((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and drive	USPAT	2003/03/04 14:53
9	214835	piston	USPAT	2003/03/04 14:54
10	116	((((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and drive) and piston	USPAT	2003/03/04 14:54

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9	214835	piston	USPAT	2003/03/04 14:54
10	116	((((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and drive) and piston	USPAT	2003/03/04 14:54

US-PAT-NO: 5902485
DOCUMENT-IDENTIFIER: US 5902485 A

TITLE: Access valve devices, their use in separation apparatus and corresponding methods

DATE-ISSUED: May 11, 1999

US-CL-CURRENT: **210/656**; **210/198.2** ; 210/281 ; **210/659**

APPL-NO: 08/ 825026

DATE FILED: March 26, 1997

PARENT-CASE:

CROSS REFERENCE TO RELATED APPLICATIONS The present invention is a continuation of PCT/GB95/02338, which has an international filing date of Oct. 3, 1995, and which is incorporated herein by reference.

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
GB	9419888	October 3, 1994

---------- KWIC ---------

Detailed Description Text - DETX:

The manifold 8 has a central bore 91 coaxial with the plate opening 31 and having inwardly and outwardly directed threaded connection openings 83, 89. The cylindrical barrel 6 of a spool valve 5 is screwed into the inward connection 83, to extend coaxially inwardly through the central plate opening 31 and out through a central circular orifice 41 of the filter layer 4, terminating in an outward flange 65 which overlaps the filter layer 4. A

cylindrical outer sleeve 66 fits snugly around the barrel 6, its outward edge
resting against the inward face of the manifold block through a polymeric
sealing ring 662 and its inner edge resting against the outer surface of the
filter layer 4 through another polymeric sealing ring 661, trapping the layer 4
between the sleeve 66 and barrel flange 65. Since the barrel's outer diameter
corresponds to that of the layer orifice 41, it is possible in the illustrated
condition to remove the barrel by unscrewing it and withdrawing it inwardly,
without disturbing the filter layer 4. This is an advantage for column
maintenance.

Detailed Description Text - DETX:

Furthermore the valve is easily dismantled for **maintenance** because the probe 7
can be entirely withdrawn inwardly from the barrel bore 61.

Detailed Description Text - DETX:

During normal running of the process the mobile phase passes through the filter
layer 4', through the filtration conduits 33' and out. There is a gradual
accumulation of particulate debris and other matter reluctant to pass the
filter 4', e.g. lipids. This therefore accumulates in an upper bed space
region 91 adjacent to filter layer 4'. In time it hinders the **maintenance** of
proper flow.

Current US Original Classification - CCOR:

210/656

Current US Cross Reference Classification - CCXR:

210/198.2

Current US Cross Reference Classification - CCXR:

210/659

	Comments	Error Definition	Errors
1			0
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7			0

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1	BRS	L1	2555	((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)	USPAT	2003/03/04 16:15
2	BRS	L2	225365	maintenance	USPAT	2003/03/04 16:15
3	BRS	L3	135	1 and 2	USPAT	2003/03/04 16:22
4	BRS	L4	3482	maintenance same screen	USPAT	2003/03/04 16:22
5	BRS	L5	0	1 and 4	USPAT	2003/03/04 16:22
6	BRS	L6	15502	remov$5 near5 screen	USPAT	2003/03/04 16:23
7	BRS	L7	13	1 and 6	USPAT	2003/03/04 16:23

	Comments	Error Definition	Errors
1			0
2			0
3			0
4			0
5			0
6			0
7			0

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3	135	(((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and maintenance	USPAT	2003/03/04 16:22
4	3482	maintenance same screen	USPAT	2003/03/04 16:22
5	0	(((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and (maintenance same screen)	USPAT	2003/03/04 16:22
6	15502	remov$5 near5 screen	USPAT	2003/03/04 16:23
7	13	(((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and (remov$5 near5 screen)	USPAT	2003/03/04 16:23
-	4484	replac$5 near5 screen	USPAT	2003/03/04 16:23
-	1683	(210/198.2).CCLS.	USPAT	2003/03/04 14:48
-	1926	(210/656-659).CCLS.	USPAT	2003/03/04 14:49
-	750	(210/635).CCLS.	USPAT	2003/03/04 14:49
-	6	(replac$5 near5 screen) and (((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.))	USPAT	2003/03/04 14:52
-	754697	drive	USPAT	2003/03/04 14:52
-	404	(((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and drive	USPAT	2003/03/04 14:53
-	214835	piston	USPAT	2003/03/04 14:54
-	116	(((((210/198.2).CCLS.) or ((210/656-659).CCLS.) or ((210/635).CCLS.)) and drive) and piston	USPAT	2003/03/04 14:54

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